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EFFLUENT WATERS

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ABSTRACT

A new flow loop in which oil/water emulsions can be recycled has been built at the University of Waterloo. The loop has five pipes of different diameters and a separate pipeline designed to receive specialized flow measuring instrumentation.

After a comprehensive literature review on the subjects of flow measurement and transport characteristics of emulsions, experimental studies have been undertaken in the areas of rheology, flow metering and pipeline flow of emulsions. Both artificial (mineral oil based) and real (heavy oil based) emulsions have been studied.

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EXECUTIVE SUMMARY

1. SCOPE OF WORK

The objectives of this work were to modify and develop a flow loop designed for the purpose of investigating pipeline flow and instrumentation of oil/water emulsions. The loop was to incorporate micro-computer data acquisition and appropriate software was to be developed. After a survey of various kinds of oils, experiments using the flow loop were to be undertaken in parallel with rheological experiments run in conventional viscometers. The flow meters were to be evaluated for their possible use as heavy oil emulsion flow meters.

2. DEVELOPMENT OF TWO PHASE FLOW METERING AND RESULTS

The apparatus development work was successfully completed so that a unique Canadian facility now exists in which experiments can be undertaken for several years to come. The loop contains pipelines of five different diameters and a return pipeline into which a broad spectrum of metering instruments can be inserted. The loop is instrumented with electrically operated pressure transducers, analog to digital converters, IBM microcomputer data acquisition equipment which has software capable of efficient data processing. It is strongly recommended that this equipment be used continuously in the coming years to generate flow data and measuring instrument data for both the Government and industry.

The flowmetering instrumentation investigated in this work was a combination of a venturi, orifice, magnetic flow meter and

conductivity meter. A set of experiments were run at various levels of concentration of Bayol/Water mixtures.

Using a mean density based on the oil/water concentration as measured by the conductivity meter, the orifice and venturi behaved in a very normal manner providing extremely good calibration curves for total mass flow. This performance extended to the situation where the emulsion inverted.

The magnetic flow meter also calibrated very well and of course had the added advantage of being independent of oil/water concentration. A drawback occurred when the phases inverted. Because the oil phase was non-conducting it was found that the magnetic flow meter gave no reading after inversion. It is recommended that the magnetic flow meter be further tested with bituminous oils because they do in fact have a conductivity and consequently, this may work in the magnetic flow meter. Certainly, the calibration curve is good when the continuous phase is conducting.

The conductance probe also calibrated for oil concentration when the continuous phase was conducting. When inversions occurred there was a sharp dip in the calibration curve making the use of the meter doubtful when the continuous phase is non-conducting. For bituminous oils this may not be the case and thus should be carefully studied.

A totally reliable and comprehensive metering system for all kinds of oil/water emulsions remains an elusive target for research engineers, although, the combination of the conductivity holdup meter with either the venturi, orifice or magnetic flow meter cannot yet be ruled out for bituminous oils.

3. EMULSION CHARACTERIZATION, RESULTS AND CONCLUSIONS

The complex problem of oil emulsion rheology was addressed and as many questions were posed by the results of the work as were answered. For example the question of whether or not conventional instruments can produce truly representative viscosity measurements arose as a result of the Weissenberg Rheogoniometer work. A conclusion reached was that it is better to use the Fann or Brookfield instrument (concentric cylinder types) to avoid interfacial slip effects.

A potentially very useful correlation was developed relating the viscosity and oil concentration of a specific emulsion species. The correlation allows a quick calculation of the viscosity of a wide range of concentrations of emulsions from one viscometer reading.

The pipeline flow characterization measurements indicated that for unstable emulsions at least, the oil/water system behaved like a Newtonian fluid in turbulent flow and a Blasius type correlation could be used to correlate the data provided the effective viscosity was used.

The following specific conclusions were reached as a result of rheological experimentation on five oil/water emulsions using three different viscometers namely the Weissenberg Rheogoniometer, the Fann Viscometer and the Brookfield viscometer.

- a) The Rheology of heavy oil dispersions is not well known or understood. There is little information in the published literature relating the Rheological properties of emulsions to their pressure drop behaviours in pipelines.

- b) Preliminary studies of a mineral oil/water emulsion using the Weissenbejrg Rheogoniometer produced encouraging results. It was initially thought that the data would correlate well by using the so-called "maximum attainable concentration" as a parameter.
- c) Subsequent work using two other kinds of viscometers, namely the Fann and Brookfield types showed disparity in the data obtained from the three instruments. It was concluded that slip effects were affecting the correct results obtained from the Rheogoniometer and in subsequent work, the use of this instrument was abandoned.
- d) A broad spectrum of Rheological data was correlated using a new parameter $\phi_{\mu r=100}$ which is the concentration at which an emulsion viscosity becomes one hundred times that of the continuous phase. It is recommended that this correlation be tested on a wide variety of oil/water emulsions of both synthetic and produced fluids and that this method be used to predict the performance of emulsion pipelines.
- e) The Rheology of a real heavy oil (supplied by Petro-Canada from its facility at Cold Lake Alberta) was investigated using the Fann and Brookfield instruments. Based on the experimental data generated, the following conclusions were made:
 - The heavy oil alone behaves like a Newtonian fluid but changes viscosity with aging.
 - The O/W emulsion has a much higher viscosity than either of the single phases.

- On aging, the emulsions exhibit thixotropic behaviour.
- The viscosity of the emulsions changes considerably with the addition of sodium chloride.
- The effect of temperature is also to reduce the viscosity of the emulsion.

Further work needs to be done to establish generalized theories and practices for the classification and standardization of heavy oil emulsions. The effects of aging, for example, are very complex and difficult to predict.

Chapter 1

INTRODUCTION

Emulsions are mixtures of two immiscible liquids, one of them being dispersed in the other in the form of globules. There may exist either an oil-in-water (oil is the dispersed phase) or water-in-oil (water is the dispersed phase) emulsion. These emulsion types are conveniently abbreviated O/W and W/O, respectively. O/W and W/O emulsions are also sometimes sub-classified as being either "stable" or "unstable". Usually stable emulsions are formed with the aid of "emulsifiers" (surface active agents) or, as in the case of natural emulsions, with the help of salts and contaminants which also have significant surface active effects. Stable emulsions do not breakdown rapidly so that the phases separate. Unstable emulsions are usually formed from mixtures of pure substances (e.g. mineral oil and distilled water). The phases rapidly separate under the influence of gravity.

1.1 Naturally Produced Emulsions

A considerable amount of the world's crude oil is produced in the form of stable emulsions (1-7). In primary oil production, when a hole is drilled through the overlying impervious strata and the pressure on the reservoir is relieved in the immediate vicinity of the well bore, the higher pressure in the other portions of the reservoir causes the fluids to migrate to the bore hole. In so doing, they are forced through fine passages or pores in the rock, sometimes at considerable velocities, and ample opportunity is provided for the oil

and water to mix. After the oil-in-water mixture has reached the well bore, it may still encounter emulsion-producing conditions as it passes through perforations in the drill pipe, through chokes, valves and other portions of the piping system. Among the types of compounds suspected of causing emulsification in primary recovery are oil-soluble organic acids such as long chain fatty acids, or esters, polycyclics, porphorins and unsaturates. Besides these active compounds, finely divided solids may be present. Such solids may be carbonaceous, polymers, clays or other materials such as elemental sulphur, iron sulphide, etc. In addition, groundwater salts have strong surface active effects enhancing the stability of emulsions in produced fluids.

In secondary oil production, water is injected into the formation through injection wells in such a way as to produce a stable front which will sweep the oil ahead of it to the producing wells. Often, the water front over runs some of the oil and breaks through into the producing wells before complete recovery is obtained. This channelling and breakthrough frequently leads to emulsion formation.

In tertiary oil recovery (surfactant flooding, polymer flooding, surfactant-polymer flooding, steam injection and caustic flooding), production from the reservoir is again, very often, in the form of a stable emulsion.

Both types of emulsions (O/W and W/O) are encountered in oil-field situations discussed above. However, water-in-oil types are encountered more frequently, containing anywhere from 0.5% to 50% water.

Many situations exist where these emulsions have to be transported long distances by pipeline from production centres to dehydration centres etc. Accurate metering and prediction of flow behaviour of these emulsions is, therefore, extremely important for royalty calculations, estimation of the capacity of existing oil pipelines and design of new pipelines.

1.2 Deliberately Produced Emulsions

The high viscosity of heavy oils or produced W/O emulsions frequently limits the rate at which crude oil can be produced from a well. It causes low pump volumetric efficiency, slow rod drop rate and high flow line pressure drop. To overcome these problems (8-12), aqueous solutions of surface active chemicals (non-ionic surfactants or soaps) are sometimes added directly to the well bore to convert highly viscous oil or W/O emulsion to low viscosity O/W emulsion.

The transportation of highly viscous crude oils (especially high pour point waxy crudes) in the form of concentrated O/W emulsions has also received special attention in the recent years (13-30). The viscosity of crude oils is greatly reduced by forming O/W emulsions. For example, the viscosity of pure Boscan crude is 80,000 cp. at 20°C. However, the viscosity of an emulsion containing 75 percent Boscan crude and 25 percent water is only 50 cp. at 20°C. Emulsion transport eliminates the need to keep viscous crude hot during transport and avoids the problem of "freeze-up" in cases of flow stoppage. Another important application for emulsion pumping is in

the Arctic and sub-Arctic regions where crude oils and tars, such as those obtained from the Athabaska tar sands, can be efficiently transported through buried pipelines at temperatures lower than 0°C in the form of 40 to 70 percent oil-in-brine emulsions. Transportation at sub-freezing temperatures eliminates serious environmental problems due to thawing of permafrost and the necessity of building an insulated pipeline suspended above the ground.

Two commercial emulsion pipeline systems for crude transport are known. One in Indonesia (15) carries 40,000 barrels/day of a 70 percent (volume) suspension of a high pour waxy crude in a 20 inch diameter line which is 238 km long. The other, in California (30), carries a 50 percent emulsion of a highly viscous 13.5 API gravity crude thirteen miles in an 8-inch pipeline.

Although these emulsions (both O/W and W/O types) are widely encountered in oil-wells, collection pipelines and transportation pipelines, the rheology, morphology and flow measurement of such emulsions are not well understood. Much of the knowledge of the behaviour of such emulsions is proprietary to the various oil companies. The flow metering problem in particular, is costing the companies a great deal at the present time. The licensing agencies require accurate oil flow measurements for royalty calculations, and the only currently acceptable method seems to be by phase separation. The capital cost of such a measurement is sometimes quoted as \$1 million per steam injection pad. Thus there is a great incentive to solve that problem.

The work described in this report represents the beginnings of an attempt to address the problems of O/W and W/O emulsion rheological classification as well as the problem of O/W and W/O emulsion metering. During the course of the work the immense complexity of the emulsion problem has become more and more apparent to the investigators. Many questions remain to be posed and answered. Some new ideas for generalizing the rheological results have been suggested and a possible system of instrumentation has been investigated.

At the outset of the work the following "tasks" were envisaged:

1. Modification and assembly of flow loop.
2. Development of a micro-computer data acquisition system for flow loop.
3. Development of data acquisition software.
4. Survey and acquisition of representative oils.
5. Running of experiments - rheological and flow loop.
6. Evaluation of experiments and flow meters.
7. Development of multiphase flow meters.
8. Final report.

This report contains results pertaining to all of the above tasks. At the beginning of each chapter in the report there is an indication as to which of the above tasks are being reported either in whole or in part.

Chapter 2

LITERATURE REVIEW

The literature reviewed in this Chapter relates to the work done in Tasks 4, 5, 6 and 7 of the contract.

2.1 Flow Measurement

Most of the previous work in this area has been restricted to W/O emulsions and numerous patents (31-42) have been issued to protect technology of this kind. The only metering system for W/O emulsions reported in the literature is due to Pal and Rhodes (43). Their metering system consists of a conductivity meter coupled with a venturi. The conductivity meter measures the oil hold-up and venturi measures the total mass flow rate of emulsion.

A detailed review of various methods of metering W/O and O/W emulsions is given in Appendix I, Papers 1 and 2. The techniques discussed are: (a) Capacitance probe coupled with a turbine flowmeter (b) Vortex meter coupled with a turbine flowmeter (c) Differential pressure unit coupled with a turbine flow meter (d) Densitometer probe coupled with a turbine flow meter and (e) Conductivity meter coupled with a venturimeter.

2.2 Transport Characteristics of Emulsions

Numerous studies have been published in recent years on multiphase flow through pipes. Fluids used have varied from a gas and a liquid to two immiscible liquids. Most previous work in the area of two phase liquid-liquid flow is restricted to the study of:

- a) Flow regimes in the flow of oil-water mixtures (44-46).
- b) Hold up and pressure losses in the flow of unstable oil-water mixtures (46-55).

Little published information is available on turbulent flow behaviour of stable O/W emulsions. Only one reference (30) is available in which studies on turbulent flow of stable O/W emulsions is reported. However, the results of this study have not been correlated successfully. Cengel, et. al (56) studied laminar and turbulent flow of dilute (oil concentration less than 50 percent) unstable oil-in-water emulsions. Assuming that the dispersion behaved as a single phase fluid, effective viscosities of the mixtures were calculated from measured friction factors. In laminar flow, viscosities were found to be a function of capillary diameter, possibly due to the formation of a thin layer of continuous phase adjacent to the capillary wall. Up to 20% oil, the viscosity of the dispersions was the same in laminar, vertical turbulent and horizontal turbulent flow. In vertical turbulent flow, all dispersions behaved as Newtonian fluids. The 35 and 50 percent dispersions in horizontal flow exhibited non-Newtonian characteristics and had effective viscosities considerably less than the same dispersion in vertical flow. The apparent non-Newtonian behaviour could be due to phase separation in the horizontal flow.

Zakin et. al, (30) studied the turbulent flow behaviour of O/W emulsions stabilized with Triton X-114 (non-ionic emulsifier). Turbulent flow pressure drop measurements were made for various concentrated O/W emulsions which exhibited non-Newtonian behaviour. Measured friction factors consistently fell below those predicted by the Dodge and Metzner friction factor correlation. No further attempt was made to correlate the friction factor data. It was suggested that the departure from the Dodge and Metzner correlation could be due to:

- a) Rheological data not representative of actual operating conditions: Rheological data (wall shear stress versus $8\bar{V}/D$) of the emulsions were obtained by use of capillary tubes. It should be noted that K' and n' vary significantly with "agitation". Samples upstream and downstream of a centrifugal pump would yield significantly different values of K' and n' .
- b) "Slip effects" near the tube wall: In laminar flow of emulsions, migration of dispersed phase particles away from the tube wall may occur. This will lead to an effective slip velocity near the tube wall. Different diameter capillaries will then give different values of n' and K' .
- c) Viscoelastic effects: It is well known that drag reducing fluids do not follow the Dodge-Metzner correlation, and these emulsions appear to be acting like them. Drag reduction observed in polymer and soap solutions is generally believed to be associated with the viscoelastic character of these solutions.

Chapter 3

FUNDAMENTALS OF NON-NEWTONIAN FLOW

The theory reviewed in this Chapter relates to the results obtained in Tasks 4 and 5 of the contract.

3.1 Steady-State Laminar Flow of Time-Independent Non-Newtonian Fluids (57-63)

A perfectly general relationship for the steady, laminar flow of any time-independent purely viscous fluid may be developed from a generalized constitutive equation. The resulting equations are usually referred to as Rabinowitsch-Mooney relations.

We start from a constitutive equation which, however complex, may be expressed as

$$f(\tau) = \gamma \quad (3.10)$$

where

τ = shear stress

γ = absolute value of shear rate = $-du/dr$ in a circular pipe.

For steady flow in a pipe

$$Q = 2\pi \int_0^R u r dr \quad (3.11)$$

where

Q = total volumetric flow rate

u = velocity at radius r

R = radius of the pipe.

Eqn. (3.11) may be expressed as

$$Q = \pi [ur^2 - \int_0^R r^2 du] \quad (3.12)$$

since $u = 0$ when $r = R$ (no slip at the wall), this simplifies to

$$Q = -\pi \int_0^R r^2 du \quad (3.13)$$

A simple force balance on a cylindrical element of radius r and length dL yields

$$-\pi r^2 dp = 2\pi r \tau dL \quad (3.14)$$

from which

$$\tau = \frac{-r}{2} \frac{dp}{dL} \quad (3.15)$$

and when $r = R$

$$-\pi R^2 dp = 2\pi R \tau_w dL \quad (3.16)$$

where τ and τ_w denote the shear stress at radius r and R , respectively. From equation (3.16) we can show that

$$r^2 = \frac{R^2}{\tau_w^2} \tau^2 \quad \text{and} \quad dr = \frac{R}{\tau_w} d\tau$$

Combining Eqs. (3.10) and (3.13) and substituting the foregoing for r^2 and dr , we obtain

$$Q = \pi \int_0^{\tau_w} \frac{R^3 \tau^2}{\tau_w^3} f(\tau) d\tau \quad (3.17)$$

If the value of τ_w is inserted in equation (3.17) and R is replaced by $D/2$, the following equation results.

$$\frac{8Q}{\pi D^3} = \frac{2V}{D} = \frac{1}{\left(\frac{D\Delta P}{4L}\right)^3} \int_0^{\frac{D\Delta P}{4L}} \tau^2 f(\tau) d\tau \quad (3.18)$$

Where L denotes the length of the pipe. This is a perfectly general relationship of the discharge Q , the pressure gradient $\Delta P/L$, the diameter D , and the constitutive equation $f(\tau) = -du/dr$. Equation (3.18) may be rearranged and differentiated with respect to $D\Delta P/4L$ to yield

$$f(\tau_w) = - \left(\frac{du}{dr}\right)_w = 3 \left(\frac{8Q}{\pi D^3}\right) + \frac{D\Delta P}{4L} \frac{d \left(\frac{8Q}{\pi D^3}\right)}{d \left(\frac{D\Delta P}{4L}\right)} \quad (3.19)$$

which is a general relationship for the rate of shear at the wall. Eqn. (3.19) is more generally written as

$$-(\frac{du}{dr})_w = \frac{8\bar{V}}{D} \left(\frac{1 + 3n'}{4n'} \right) \quad (3.110)$$

where

$$n' = \frac{\frac{D\Delta P}{4L}}{\frac{d \ln \left(\frac{8\bar{V}}{D} \right)}{d \ln \left(\frac{D\Delta P}{4L} \right)}} \quad (3.111)$$

From the definition of n' (Eqn. 3.111), it is easily seen that we can write

$$\tau_w = \frac{D\Delta P}{4L} = K' \left(\frac{8\bar{V}}{D} \right)^{n'} \quad (3.112)$$

This is the equation of the tangent to the logarithmic plot of τ_w and $8\bar{V}/D$ at a chosen value of τ_w , and K' corresponds to a particular value of τ_w . From the logarithmic plot of $D\Delta P/4L$ against $8\bar{V}/D$ we can find n' . The corresponding shear rate at the wall then follows from Eqn. (3.110). In this way we can determine the shear stress and rate of shear at corresponding points, and the flow curve (τ vs γ) can

be drawn by repeating this procedure at different values of the wall shear stress. If the slope of the logarithmic plot is constant over a range of shear stresses, Eqn. (3.112) represents the equation of this straight line. If the plot is not a straight line, n' and K' must be evaluated at the appropriate shear stress.

For a power law pseudoplastic, it can be easily shown that

$$n' = n \quad k' = k \frac{(1 + 3n)n}{n} \quad (3.113)$$

Where n and K are power law constants ($\tau = K \dot{\gamma}^n$).

Friction Factor Versus Reynolds Number for Non-Newtonian Fluids

The friction factor, f , is defined in the usual manner by

$$f = \frac{\tau_w}{1/2(\rho V^2)} = \frac{D\Delta p}{4L} / \frac{\rho \bar{V}^2}{2} \quad (3.114)$$

In order to be able to use the conventional chart for Newtonian fluids and non-Newtonian fluids alike in the laminar region, it is convenient to let $f = 16/N'Re$ as for Newtonians in laminar flow. Since

$$\tau_w = \frac{D\Delta p}{4L} = K' \left(\frac{8\bar{V}}{D} \right)^{n'} \quad (3.112)$$

$$f = K' \left(\frac{8\bar{V}}{D} \right)^{n'} / \frac{\rho \bar{V}^2}{2} \quad (3.115)$$

Combining Eqn. (3.115) and $f = 16/N'Re$ gives the generalized Reynolds number.

$$N'Re = \frac{D^{n'} \bar{V}^{2-n'} \rho}{K' 8^{n'-1}} \quad (3.116)$$

$f = 16/N'Re$ ($N'Re$ defined by Eqn. (3.116)) then represents a generalized relationship applicable to the laminar pipe flow of any time-independent fluid. Metzner and Reed (63) indicate that stable laminar flow of time independent non-Newtonian fluids extends to a value of $N'Re$ of 2000 to 2500.

3.2 "Slip Effects" in Steady State Laminar Flow of Time-Independent Non-Newtonian Fluids (15, 56, 64-79)

The movement of particles away from the tube wall, due to preferred orientation of particles caused by the presence of the wall, enables the generation of a low viscosity layer and this gives rise to an effective slip velocity at the wall. This effect, for example, has been shown with a suspension of paper pulp, i.e. when the material flows through a transparent pipe, a clear water annulus is observed adjacent to the wall (65-67). The experimental verification of particle migration away from the tube wall in a variety of other multiparticle suspension flows has been reported by several authors (74-79).

If there is no anomalous behaviour or effective slip near the tube wall, $Q/\pi R^3$ versus $D\Delta P/4L$ data will result in a single curve for different tube diameters. However, if there is effective slip near the tube wall, the curves will be distinct for each value of R .

3.3 Steady-State Turbulent Flow of Time-Independent Non-Newtonian Fluids (80-93)

Dodge and Metzner (80) carried out a semi-theoretical analysis of turbulent flow in smooth tubes of time-independent viscous non-Newtonian fluids described by the power law. Applying the techniques of dimensional analysis for the power law fluid, as Millikan (81) has done for Newtonian fluids, they derived the expression

$$\frac{1}{\sqrt{f}} = A_{1n} \log [N'_{Re} f^{\frac{(1-n')}{2}}] + C'_n \quad (3.30)$$

where

$$C'_n = A_{1n} \log \left[\frac{1}{8} \left(\frac{3 + 6n'}{n'} \right)^{n'} \right] + C_n$$

The terms A_{1n} and C'_n are functions of n' to be determined experimentally. If n' is taken as unity, Eqn. (3.30) reduces to

$$\frac{1}{\sqrt{f}} = A \log \left(\frac{\rho D V}{\mu} \sqrt{f} \right) + C \quad (3.31)$$

which is of the same form as the Nikuradse equation, with $A = 4.0$ and $C = -0.40$. For the power law fluids which they tested, Dodge and Metzner found empirically that

$$A_n = \frac{4.0}{(n')^{0.75}} \quad (3.32)$$

and

$$C'_n = \frac{-0.4}{(n')^{1.2}} \quad (3.33)$$

Incorporating these values into eqn. (3.30), we obtain

$$\frac{1}{\sqrt{f}} = \frac{4.0}{(n')^{0.75}} \log [N'_{Re} f^{\frac{(1-n')}{2}}] - \frac{0.40}{(n')^{1.2}} \quad (3.34)$$

This relationship is reflected in graphical form in Figure (3.1). The authors indicate excellent agreement between calculated and experimental friction factors over values of n' from 0.36 to 1.0 and N'_{Re} from 2900 to 36,000 with solid-liquid suspensions and polymeric solutions. They show that non-power law clay suspensions also conform

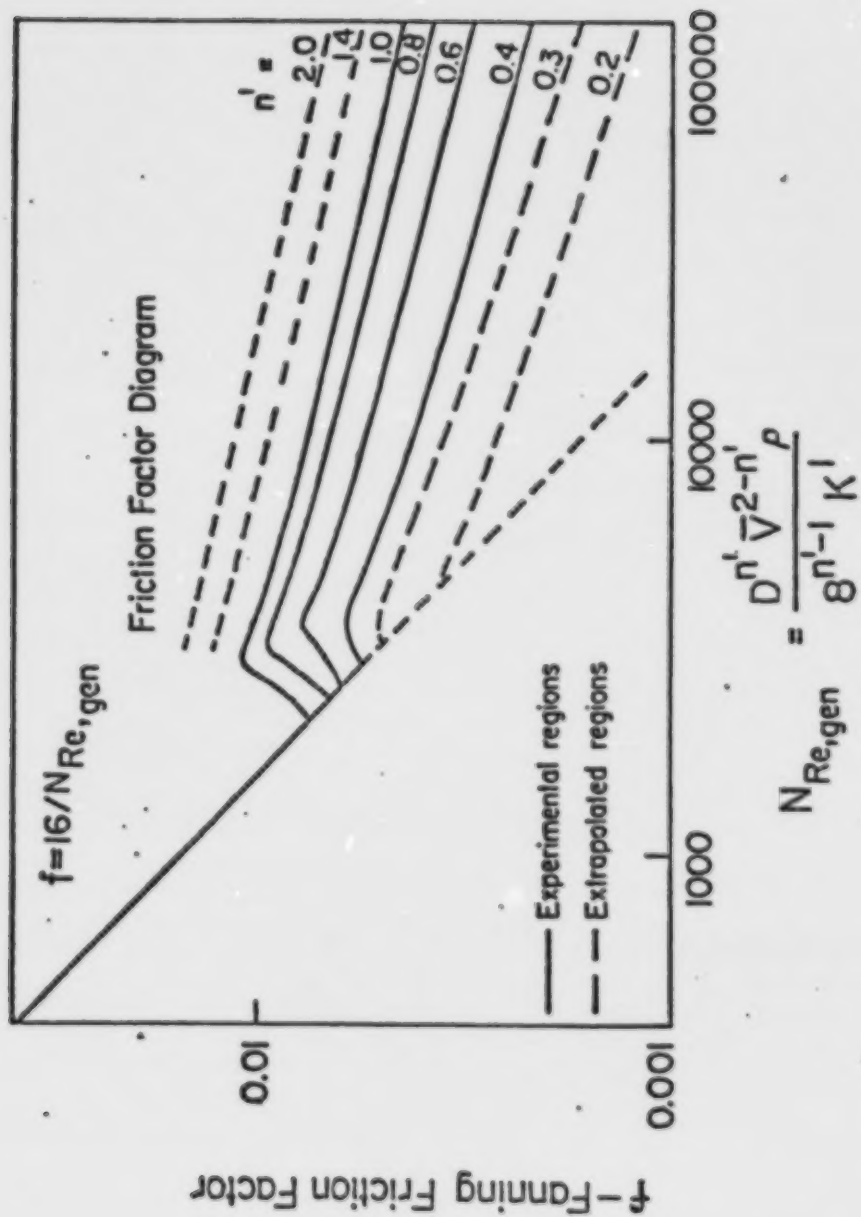


Figure 3.1: Friction Factor Diagram for Non-Newtonian Flow as Suggested by Dodge and Metzner (80).

to the correlation provided that the exponent n' is evaluated at a stress corresponding with the wall shear stress.

3.31 Other Relations for Turbulent Flow: It has usually been pointed out that the apparent viscosity of many non-Newtonian fluids under turbulent flow conditions is nearly constant and, as a result, the conventional diagram relating friction factor and Reynolds number which has been developed for Newtonian systems is also applicable to non-Newtonian systems if a realistic value is chosen for the viscosity term in the Reynolds number. The basis for this approach is the well-known fact that non-Newtonian fluids of the pseudoplastic and Bingham plastic types approach a limiting viscosity at sufficiently high rates of shear and thereafter appear Newtonian.

Caldwell and Babbitt (84) in their work on sewage sludges and slurries used the viscosity of the dispersion medium. The basis of this assumption is the fact that with increasing shearing rate the apparent viscosity of dispersions decreases and approaches that of the dispersion medium.

Other workers, notably Alves, Boucher and Pigford (85) and also Winding et al. (86), have suggested that the limiting viscosity μ_{∞} at an infinite shear rate is more realistic. These workers found that the conventional plot of friction factor against Reynolds number for Newtonian systems correlated the results for non-Newtonian systems satisfactorily if the Reynolds number is defined as $\rho DV/\mu_{\infty}$.

Another approach which has advantages over the method using μ_{∞} involves the use of a "turbulent viscosity". This is found from the relation between pressure drop and flow rate for the particular fluid under turbulent conditions. The experimentally determined friction

factor so obtained is used to calculate the Reynolds number from the Newtonian friction factor chart. This Reynolds number is equated to $\rho \bar{V} D / \nu_t$ to give ν_t , the "turbulent viscosity". This is then assumed constant. It is clear that the disadvantage of this approach is that it does not allow the design of a pipeline directly from a knowledge of flow properties. It is in fact a scale-up procedure whereby full-scale equipment can be designed once the empirical "turbulent viscosity" has been determined for a given fluid in a similar pilot plant. Alves et al. have adopted this approach (85).

The drawback of the methods discussed above is that they do not take into account the possibility of non-Newtonian behaviour prevailing under the conditions of shear existing in turbulent flow. However, the semi-theoretical analysis of Dodge-Metzner, partially takes into account the non-Newtonian behaviour prevailing under the conditions of turbulent flow.

3.32 Scale-Up Procedures

3.321 Bowen's Method (89): Bowen has proposed a scale-up method which seems to work for the turbulent flow of any time-independent viscous or viscoelastic fluid (or drag reducing fluid) or suspension which exhibits a linear relationship in a logarithmic plot of $D\Delta P/4L$ versus $8\bar{V}/D$ under turbulent conditions. A nearly straight parallel family of curves is obtained when the turbulent flow data are plotted ($\log D\Delta P/4L$ vs $\log 8\bar{V}/D$) for different pipe diameters.

A similar family of nearly straight parallel curves results when turbulent flow data obtained with Newtonian fluids are plotted in the

same manner. This suggests that an equation form for this family may also be derived from turbulent flow conditions.

Substitution of the Fanning friction factor,

$$f = \frac{D\Delta p}{2L\rho\bar{V}^2} \quad (3.35)$$

and the Reynolds number

$$N_{Re} = \frac{\rho D\bar{V}}{\mu} \quad (3.36)$$

into the Blasius expression

$$f = \frac{(0.079)}{(N_{Re})^{0.25}} \quad (3.37)$$

yields:

$$\frac{D\Delta p}{4L} = \frac{0.079 (\rho)^{0.75} (\mu)^{0.25} (D)^{1.5}}{2(8)^{1.75}} \left[\frac{8\bar{V}}{D} \right]^{1.75} \quad (3.38)$$

This equation suggests a constant A containing fluid density and viscosity:

$$A = \frac{0.079 (\rho)^{0.75} (\mu)^{0.25}}{(2) (8)^{1.75}} \quad (3.39)$$

the diameter D raised to an exponent term e equal to 1.6 for Newtonian fluids; and the flow function raised to an exponent term s equal to 1.8 for Newtonian fluids. The following general relationship:

$$\frac{D\Delta p}{4L} = A D^e \left(\frac{8V}{D}\right)^s \quad (3.310)$$

is thus suggested for any fluid describing straight parallel lines on the logarithmic plot of flow and stress function in the turbulent region. Bowen's purely empirical method requires turbulent flow friction factor data on at least two pipe diameters to obtain the power on the diameter.

3.322 Lord, Hulsey and Melton Scale-up procedure (92-93): A generalized turbulent flow relationship derived from the Blasius expression and suggested by Bowen (eqn. 3.310) can be rearranged as

$$\frac{\Delta p}{4L} = A D^{e-s-1} (8V)^s \quad (3.311)$$

For Newtonian fluids, eqn. (3.311) can be written as

$$\frac{\Delta P}{4L} = A D^{-1.25} (\overline{8V})^{1.75} \quad (3.312)$$

The constant A contains fluid density and viscosity and is given by eqn. (3.39).

Lord et al. analyzed their data in pipes ranging from 1/8 to 1 inch in diameter to obtain values of the constants A, e, and s. The net diameter exponent (e-s-1) was found to be more or less independent of fluid properties permitting eqn. (3.311) to be rearranged as

$$\frac{D^{1.25} \Delta P}{4L} = A (\overline{8V})^s \quad (3.313)$$

With this model, data from all pipe diameters should form a single line on $\log(D^{1.25} \Delta P / 4L)$ versus $\log(\overline{8V})$ plot for each fluid. This correlation should enable one to scale up from data which represents only one diameter.

3.4 Drag Reduction (94-132)

Drag reduction is defined as the reduction in pressure drop of a solvent to which a "drag reducing" material has been added as compared to the pressure drop of the pure solvent at the same flow rate in the same tube. In mathematical terms,

$$\text{percent drag reduction} = \frac{(\Delta P_0 - \Delta P_{\text{additive}})}{\Delta P_0} \times 100 \quad (3.40)$$

Drag reduction is distinct from the decreased friction factor observed by Dodge and Metzner (80) and exhibited by purely viscous pseudoplastic fluids in turbulent flow at a given generalized Reynolds number. Friction factors for drag reducing fluids may be far below those predicted using the Dodge-Metzner correlation:

$$\frac{1}{\sqrt{f}} = \frac{4.0}{(n')^{0.75}} \log [N' \text{Re} f^{\frac{(1-n')}{2}}] - \frac{0.40}{(n')^{1.2}} \quad (3.34)$$

The drag reduction phenomenon is exhibited by many Newtonian and pseudoplastic solutions, gels, and suspensions, and is a departure from their "normal" viscous behaviour. The pseudoplastic friction factor effect correlated by Dodge and Metzner is regarded to be "normal" viscous behaviour.

Drag reduction occurs in single phase turbulent flow and may be achieved with several types of additives. Additives that have been used with success include high molecular weight polymers (94-113), solid particle suspensions (114-116), soaps (117-126), fibres (127-130) and other complex-forming mixtures that behave in a manner similar to soaps (131-132).

Chapter 4

PRINCIPLES OF FLOW MEASUREMENT

The review given in this Chapter pertains to the work done in Tasks 1, 2, 3, 5, 6 and 7 of the contract.

On-line measurement of both oil and water flow rates in an emulsion must involve two independent measurements, e.g. dispersed phase concentration and the total flow rate. These two measurements are then combined to provide the individual flow rates. In this work an on-line conductivity meter has been used to determine the dispersed phase concentration and orifice, venturi and magnetic flow meters were used to determine the total flow rate. Reviews of oil/water emulsion flow measuring techniques are given in Papers 1 and 2 of Appendix 1.

4.1 Conductivity Meter (133-139)

The electrical conductivity of an emulsion depends upon several factors (133) such as:

- i) Type of emulsion (O/W or W/O)
- ii) Conductivities of continuous and dispersed phases
- iii) Volume concentration of dispersed phase
- iv) Measuring frequency
- v) Temperature

Emulsions in which water is the continuous medium (O/W type) show a high conductivity; emulsions in which oil is the continuous medium (W/O type) may be expected to show little conductivity. The conductivity of O/W emulsions increases with the temperature and decreases with the increase in volume concentration of the oil phase.

The conductivity of W/O emulsions increases with the temperature and volume concentration of dispersed phase (i.e. water).

For a dispersion of spherical particles in a liquid at low concentrations, Wagner (134) derived the following relationship between the complex dielectric constant of the dispersion and the volume fraction:

$$\frac{\epsilon^* - \epsilon_m^*}{\epsilon^* + 2\epsilon_m^*} = \frac{\epsilon_p^* - \epsilon_m^*}{\epsilon_p^* + 2\epsilon_m^*} \quad \phi \quad (4.10)$$

Since the above equation is derived for a dilute dispersed system it does not hold for concentrated dispersions. Hanai (135-137) developed a theory for a concentrated dispersed system on the basis of Wagner's Theory. Hanai made the following assumptions.

1. A concentrated dispersed system can be obtained by successively adding infinitesimally small quantities of dispersed phase into the dispersion medium.
2. Wagner's relation (Eqn. 4.10) holds for this process with infinitesimally small change in concentration.

Based on the above assumptions, Hanai derived the following relationship:

$$\frac{\epsilon^* - \epsilon_m^*}{\epsilon_m^* - \epsilon_p^*} \left(\frac{\epsilon_m^*}{\epsilon^*} \right)^{1/3} = 1 - \phi \quad (4.11)$$

on substitution of the relations for ϵ^* , ϵ_p^* , ϵ_m^* and separating the real and imaginary parts, Eqn. (4.11) reduced to:

i) at low frequencies ($\omega \rightarrow 0$)

$$\frac{K_L - K_D}{K_M - K_P} \left(\frac{K_M}{K_L} \right)^{1/3} = 1 - \phi \quad (4.12)$$

and

$$\epsilon_L \left[\frac{3}{K_L - K_P} \right] = 3 \left[\frac{\epsilon_M - \epsilon_D}{K_M - K_P} + \frac{\epsilon_D}{K_L - K_P} \right] - \frac{\epsilon_M}{K_M} \quad (4.13)$$

ii) At high frequencies ($\omega \rightarrow \infty$)

$$\frac{\epsilon_H - \epsilon_D}{\epsilon_M - \epsilon_P} \left(\frac{\epsilon_M}{\epsilon_H} \right)^{1/3} = 1 - \phi \quad (4.14)$$

$$K_H \left[\frac{3}{\epsilon_H - \epsilon_P} - \frac{1}{\epsilon_H} \right] = 3 \left[\frac{K_M - K_D}{\epsilon_M - \epsilon_P} + \frac{K_D}{\epsilon_H - \epsilon_P} \right] \quad (4.15)$$

For O/W emulsions

$K_M \gg K_P; \epsilon_M \gg \epsilon_P; K_L \gg K_P; K_H \gg K_P$ and $\epsilon_H \gg \epsilon_P$

Eqn. (4.12) reduces to:

$$\frac{K_h}{K_m} = (1 - \phi)^{3/2} \quad (4.16)$$

Eqn. (4.14) reduces to:

$$\frac{\epsilon_h}{\epsilon_m} = (1 - \phi)^{3/2} \quad (4.17)$$

Eqn. (4.15) reduces to:

$$\frac{K_h}{K_m} = \frac{\epsilon_h}{\epsilon_m} \quad (4.18)$$

From Eqns. (4.17) and (4.18):

$$\frac{K_h}{K_m} = (1 - \phi)^{3/2} \quad (4.19)$$

From Eqns. (4.16) and (4.19):

$$K_h = K_m \quad (4.110)$$

For W/O emulsions

$$K_p \gg K_m; K_p \gg K_h; K_p \gg K_L; \epsilon_p \gg \epsilon_m; \epsilon_p \gg \epsilon_h; \epsilon_p \gg \epsilon_L$$

Eqn. (4.12) reduces to:

$$\frac{K_L}{K_m} = (1 - \phi)^{-3} \quad (4.111)$$

Eqn. (4.14) reduces to:

$$\frac{\epsilon_h}{\epsilon_m} = (1 - \phi)^{-3} \quad (4.112)$$

Eqn. (4.15) reduces to

$$\frac{K_h}{K_m} = \frac{\epsilon_h \epsilon_p}{\epsilon_m} \left(\frac{1}{\epsilon_p + 2\epsilon_h} \right) \quad (4.113)$$

4.2 Orifice and Venturimeters (Differential Pressure Meters)

The mean flow rate of a single phase fluid through a differential pressure meter is given by

$$W = \frac{C_D A_0}{\sqrt{1 - \beta^4}} \sqrt{2\rho \Delta p} \quad (4.20)$$

where

C_D = discharge coefficient which is a function of Reynolds number ($= \rho D_0 \bar{V} / \mu$)

A_0 = throat (or orifice) cross-sectional area

β = D_0/D = ratio of throat (or orifice) diameter to pipe diameter

Differential Pressure Meters for Emulsions:

If it is assumed that the emulsions behave as single phase liquids, the following equation (i.e. homogeneous flow model) can be used to determine the mean flow rate through the meter.

$$W = \frac{C_D A_0}{\sqrt{1 - \beta^4}} \sqrt{2\rho \Delta p}$$

where

ρ = mean density of the mixture

C_D = discharge coefficient

Discharge Coefficient Versus Reynolds Number

The principal difference between single phase flow and emulsion flow is the choice of viscosity to be used in the Reynolds number. The following methods will be used to correlate discharge coefficient versus Reynolds number in the present study.

(i) Viscosity of dispersion medium: Caldwell and Babbitt (84) in their work on pipeline flow of sewage sludges and slurries used the viscosity of the dispersion medium (in the Reynolds number) to

correlate turbulent flow friction data. They found that the conventional friction factor-Reynolds number chart for Newtonian fluids is also applicable to slurry flow if the dispersion medium viscosity is used in Reynolds number. Thus one would expect that C_p vs N_{Re} for Newtonian fluids could be used for emulsions if dispersion medium viscosity is used in N_{Re} .

(ii) Limiting viscosity at high shear rates: The discharge coefficient against Reynolds number for Newtonian fluids could be used for emulsions if the Reynolds number is defined as $\rho V D / \mu_{\infty}$, where μ_{∞} = viscosity at high shear rates.

(iii) Generalized Reynolds number: A generalized Reynolds number is defined as:

$$N'_{Re} = \frac{\rho D^{n'} \bar{V}^{2-n'}}{K' 8^{n'-1}} \quad (3.116)$$

K' and n' are rheological parameters determined from pipeline viscometers ($\tau_w = K' (8\bar{V}/D)^{n'}$).

The discharge coefficient for emulsions will be plotted against the generalized Reynolds number in this work.

Chapter 5

EXPERIMENTAL APPARATUS AND CALIBRATION

The experimental apparatus descriptions given in this Chapter pertain to Tasks 1, 2, 3, 5, 6 and 7 of the contract.

5.1 General Description of Flow Loop Apparatus

The emulsion flow measuring apparatus consists of a loop in which emulsions may be prepared and recycled through various sizes of pipelines and instruments. A schematic diagram and general views of the experimental apparatus are shown in Figures 5.1, 5.2 and 5.3. It consists of two centrifugal pumps (Hayward Gordon-model AA6 and Hayward Gordon model IFLA) both of them having a by-pass to regulate the flow; two-S.S. mixing tanks of capacity 260 U.S. gals. and 60 U.S. gals.; two Philadelphia portable mixers; five-S.S. straight pipe test sections; metering test section consisting of orifice plate meter, venturimeter, magnetic flow meter and a conductivity meter; a temperature controller with heating (cooling) coil in the main tank; S.S. pressure tank containing water to purge the pressure lines and taps; several valdyne transducers; manometers; various thermocouples and a measuring tank to measure the flow rates. An IBM personal computer is used to collect data from various electrical signal transducers (see below for description of the data acquisition system).

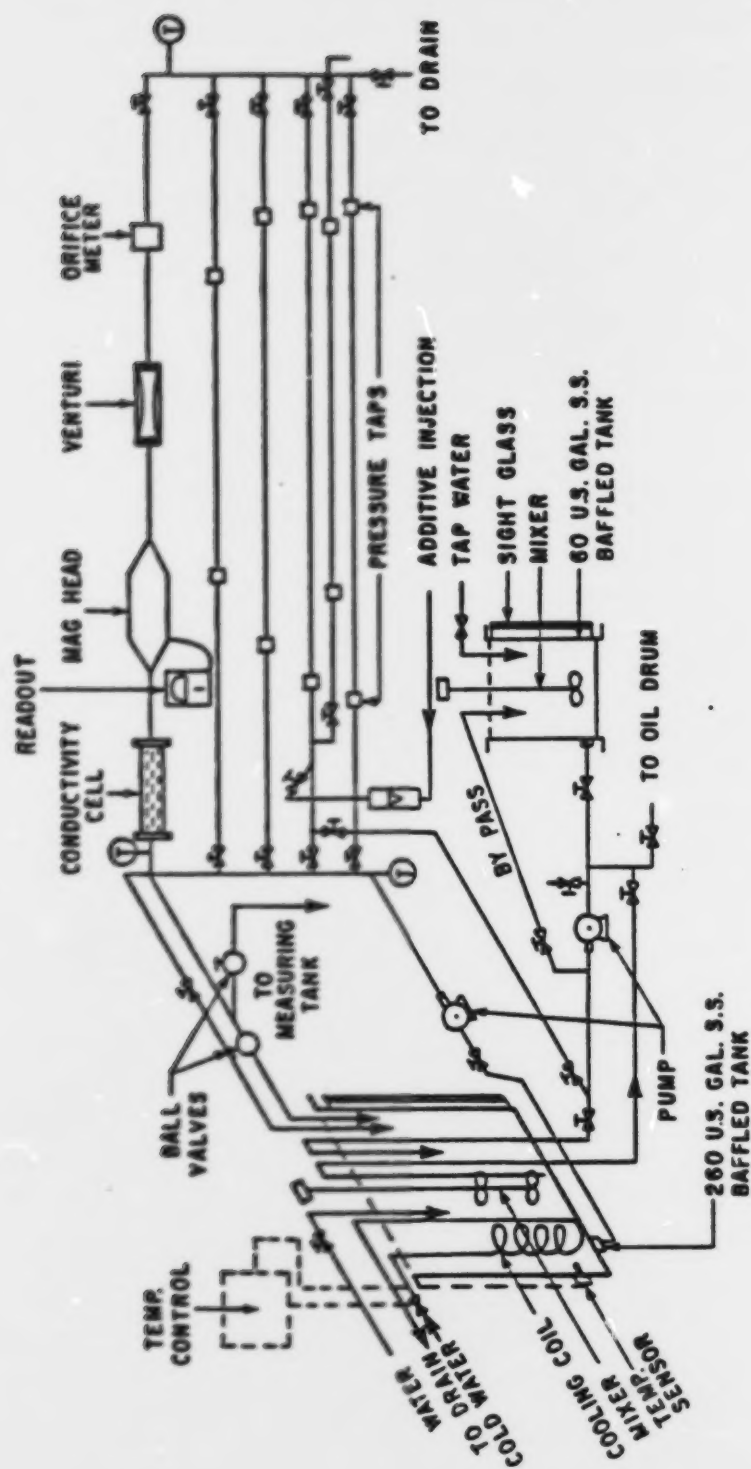


Figure 5.1: Schematic Diagram of Experimental Apparatus

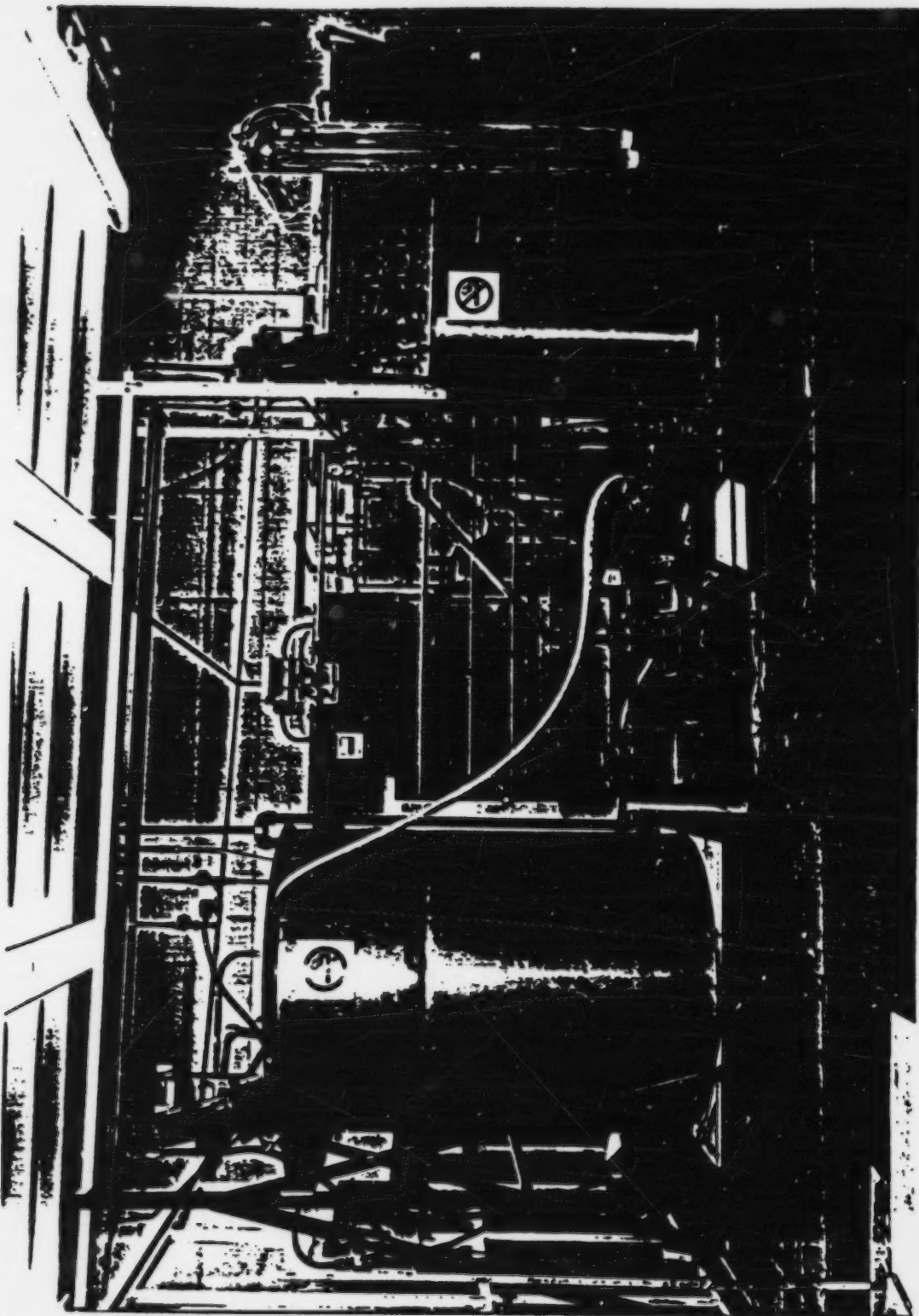


Figure 5.2: Front View of
Experimental Apparatus

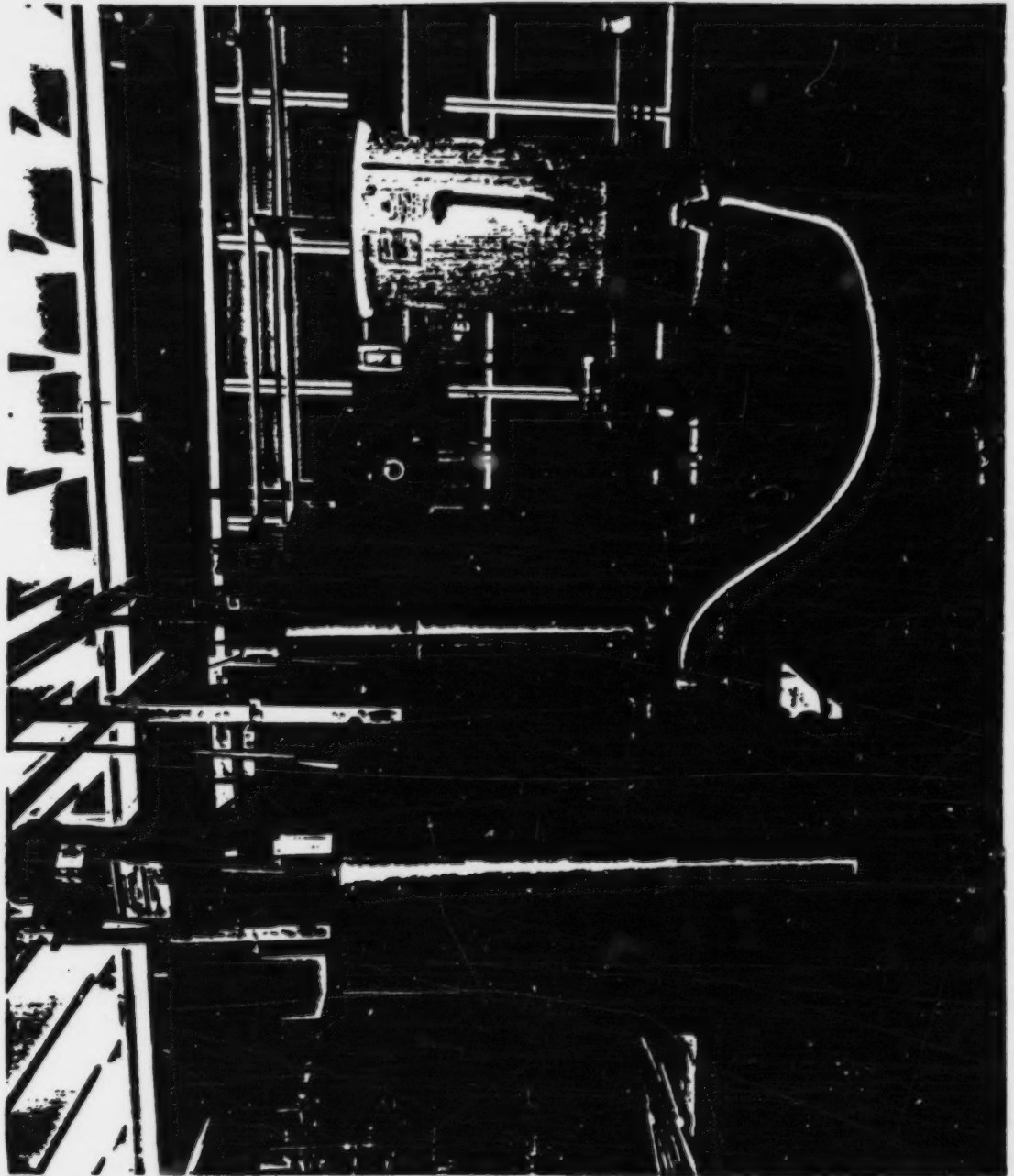


Figure 5.3: Side View of Experimental Apparatus

5.2 Straight Pipe Test-Sections

Five smooth and straight seamless pipe test sections of 1.045", 0.623", 0.496", 0.35", and 0.2815" internal diameter are provided. All the test sections are 18 ft. long and are welded at both ends to common manifolds. A drain is provided at the end of these test sections to allow drainage of the test fluid.

Table 5.1 gives the details of these test sections.

Table 5.1: Details of Pipe Test Section						
Nominal Diameter	Inside Diameter	Valve at Inlet	Entrance length	Actual length of test section	Exit length	Valve at Exit
3/8"tube	0.2815"	Needle	3.5 ft	10 ft	1.5 ft	Ball
1/4"pipe	0.35"	Needle	2.92 ft	11 ft	1.58 ft	Ball
3/8"pipe	0.496"	Ball	3.92 ft	9 ft	1.75 ft	Ball
1/2"pipe	0.623"	Needle	5.4 ft	8.5 ft	1.84 ft	Ball
1.0"pipe	1.045"	Ball	10 ft	4 ft	2.2 ft	Ball

Suitable supports are provided to hold the pipes rigid and horizontal.

The pressure taps are made by carefully drilling four holes perpendicularly through the pipe walls at 90° apart. Burrs at the inside of these taps, resulting from drilling the tapping holes, were removed with a very fine sand paper attached to a long steel shaft which was given slow rotary motion by a motor. The four holes at each

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Suitable supports are provided to hold the pipes rigid and horizontal.

The pressure taps are made by carefully drilling four holes perpendicularly through the pipe walls at 90° apart. Burrs at the inside of these taps, resulting from drilling the tapping holes, were removed with a very fine sand paper attached to a long steel shaft which was given slow rotary motion by a motor. The four holes at each

station along the circumference of the pipe are connected by a 0.3175 cm wide piezometer ring made out of transparent acrylic. (See Figs. 5.4 and 5.6 for typical rings).

5.3 Metering Test Section

The metering test section consists of an orifice plate meter, venturi meter, electromagnetic flowmeter and a conductivity meter.

a) Orifice meter: A schematic diagram of the orifice plate meter is shown in Figure 5.4. It is made in accordance with the British standards. It is installed 2.5 ft. downstream of the metering test section. The upstream pressure tap is placed a distance of one pipe diameter from the orifice plate and the downstream pressure tap is placed a distance of $D/2$ from the plate.

b) Venturi meter: A detailed diagram of the venturi meter is shown in Figure 5.5. It is installed 4.4 ft. downstream of the orifice plate and is constructed from 316-S.S. according to British standards. Two pressure taps are located on the venturi section; one just upstream of the constriction and one at the throat.

c) Electromagnetic flow meter: A Brooks MAG 7000 magnetic flow meter (Figure 5.6) is installed 1.42 ft. downstream of venturi meter. The flow head is a 1.0" stainless steel tube lined with teflon. The electrodes, which are mounted at right angles to the magnetic field, are made out of Hastelloy C-276. The signal (frequency output) from the flow head is relayed to a panel mounted frequency-to-analog converter with dial read out.

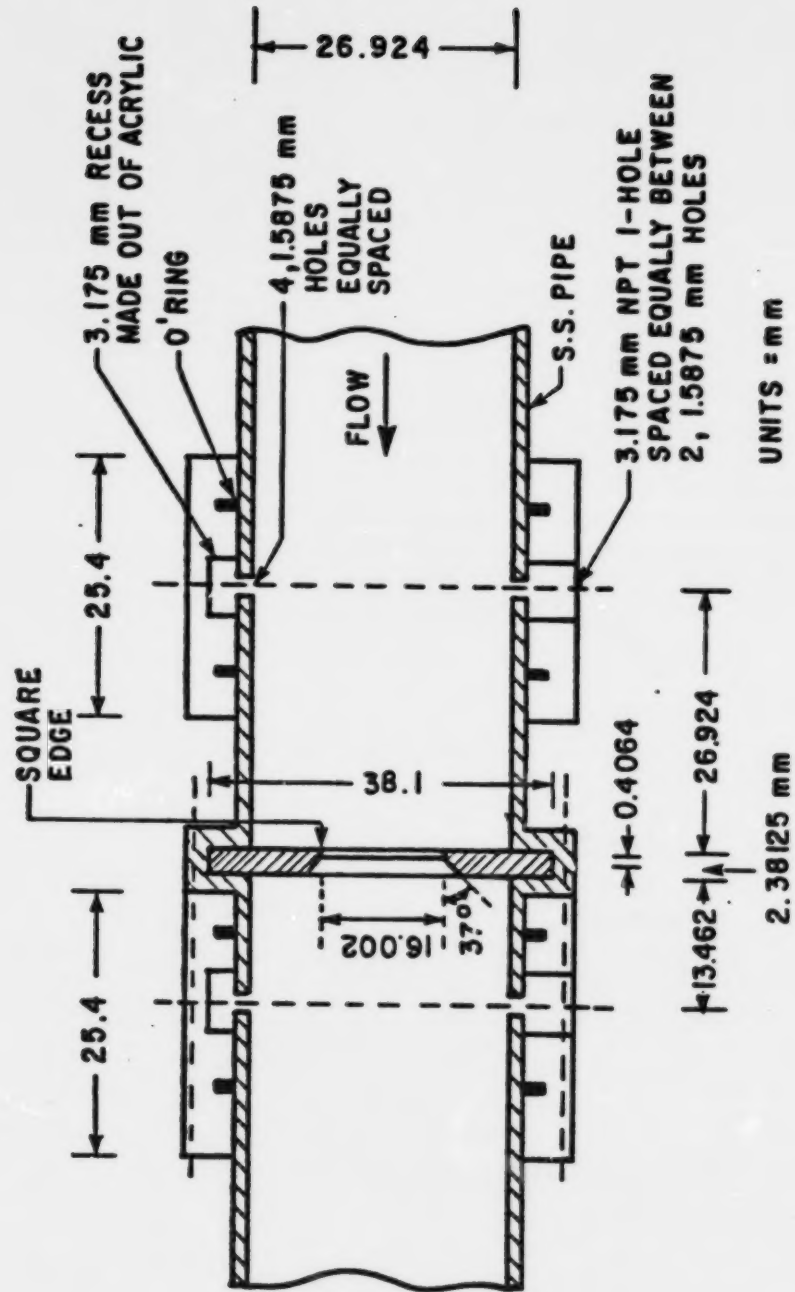


Figure 5.4: Schematic Diagram of Orifice Plate Meter

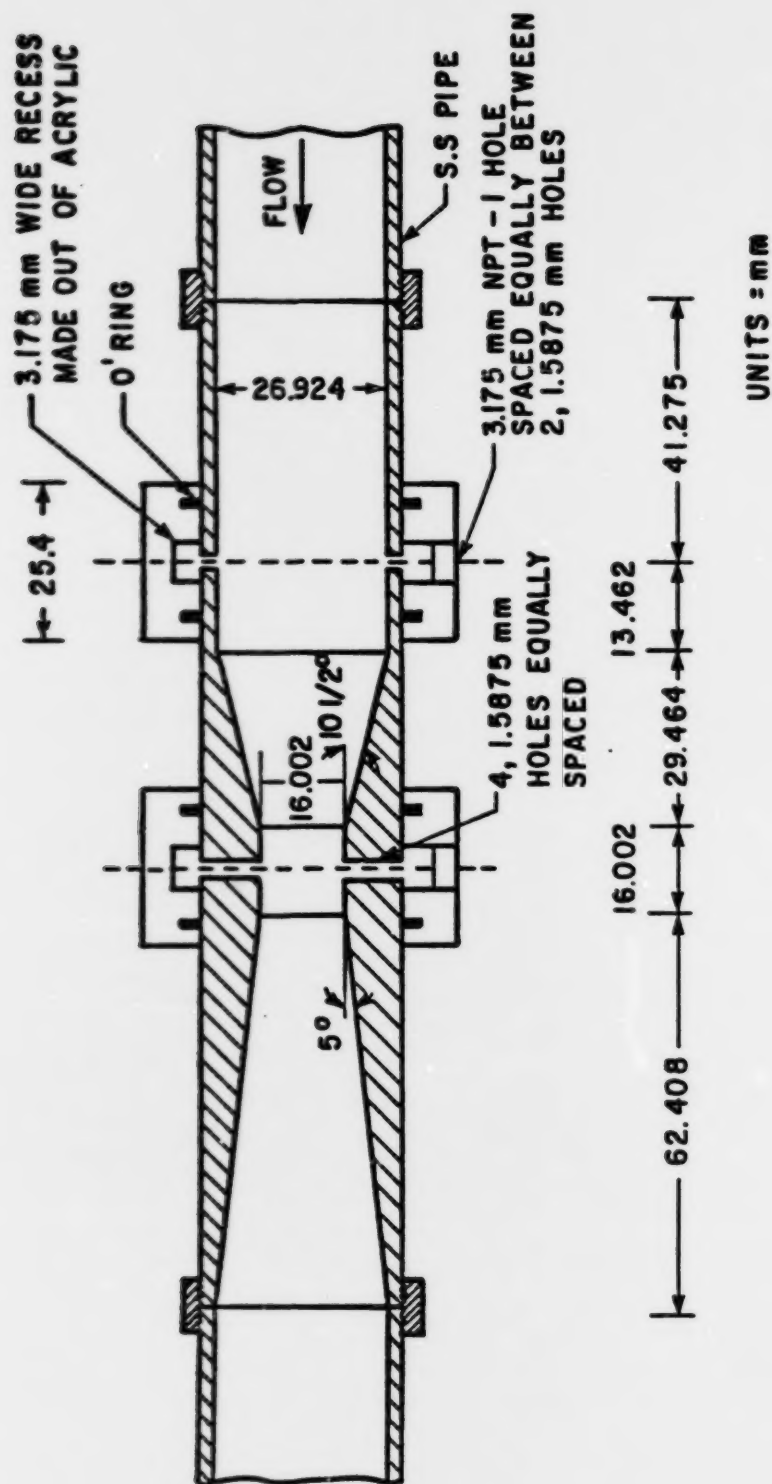


Figure 5.5: Schematic Diagram of Venturi Meter

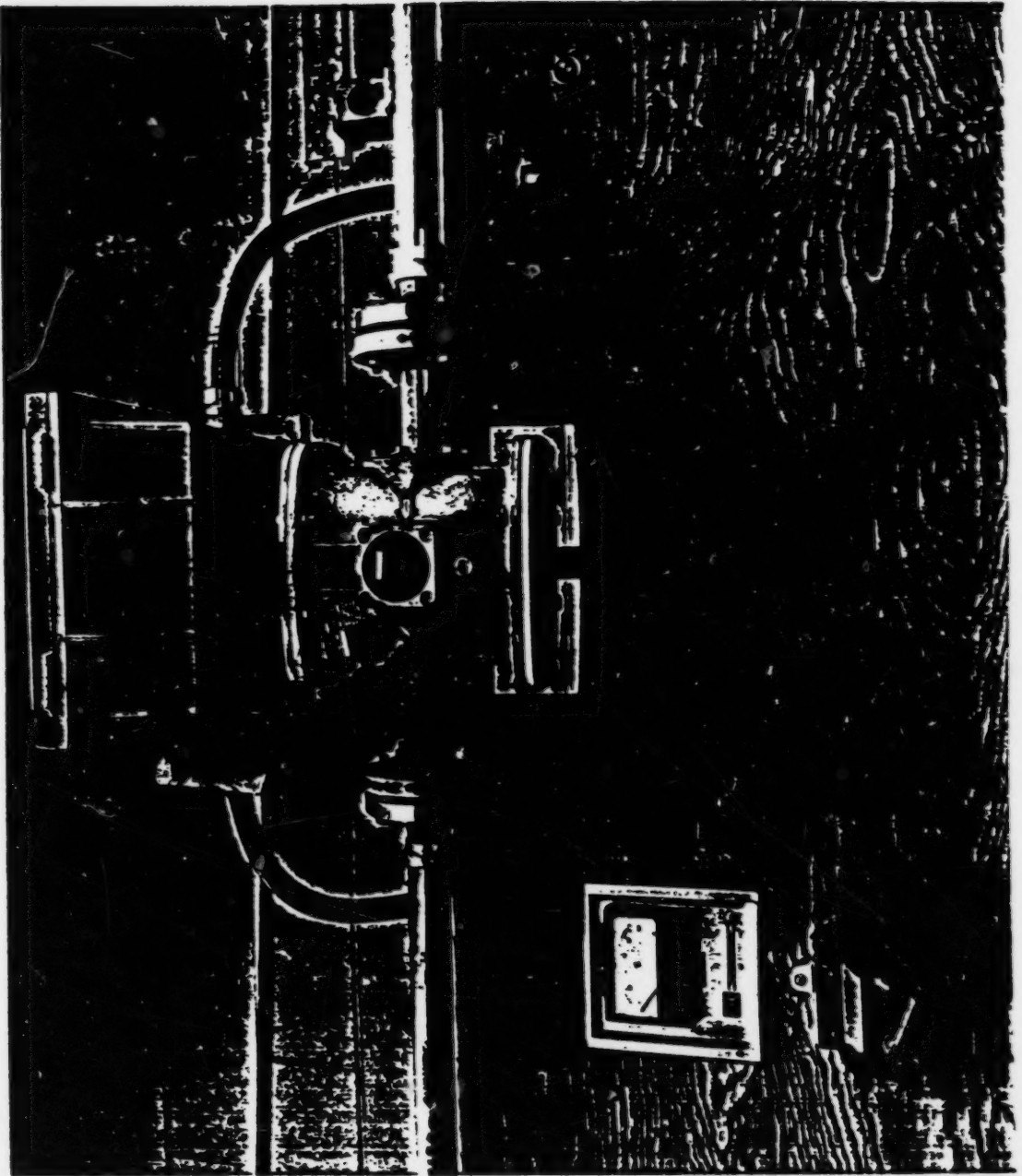


Figure 5.6: Magnetic Flow Meter

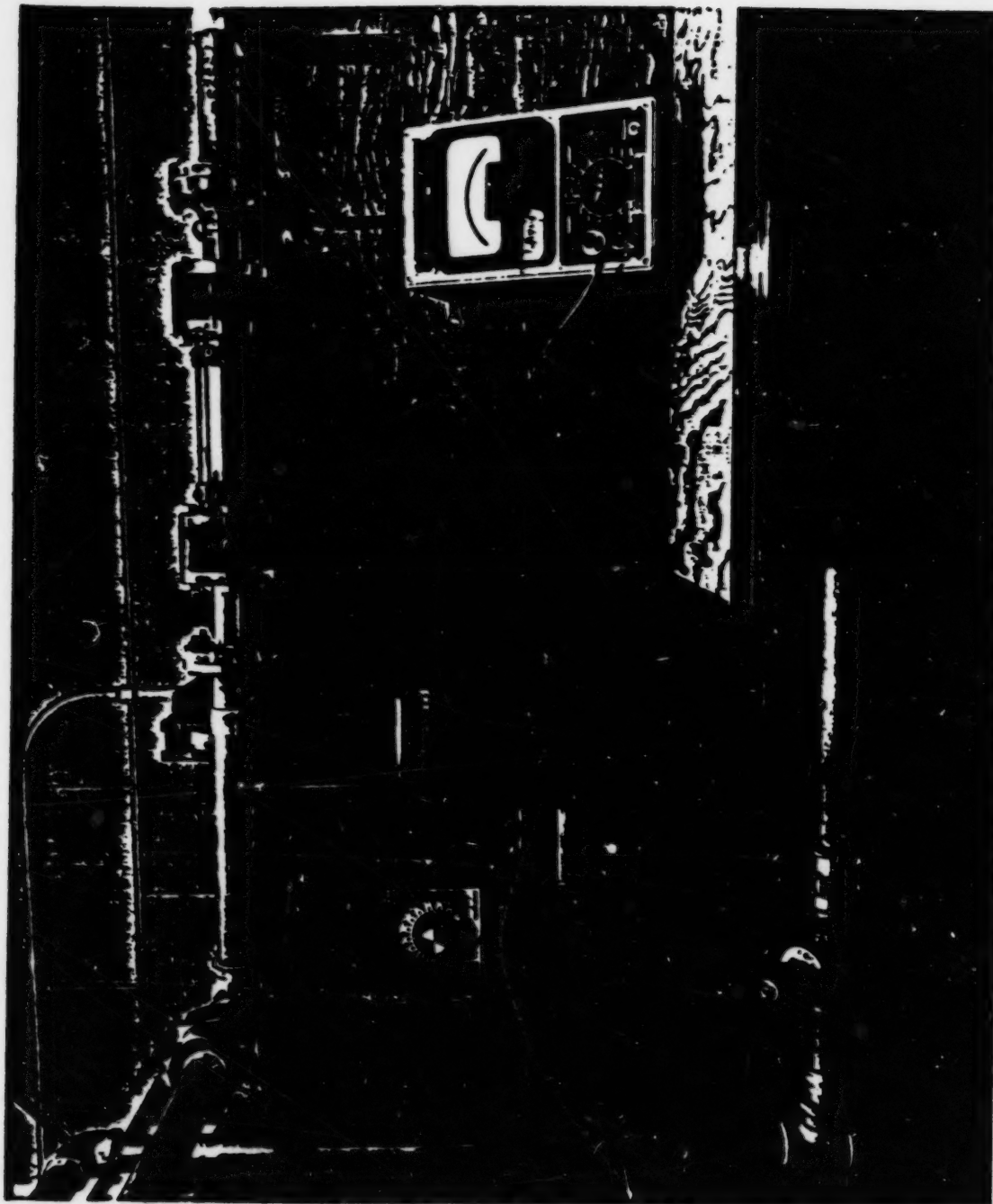


Figure 5.7: Conductivity Meter

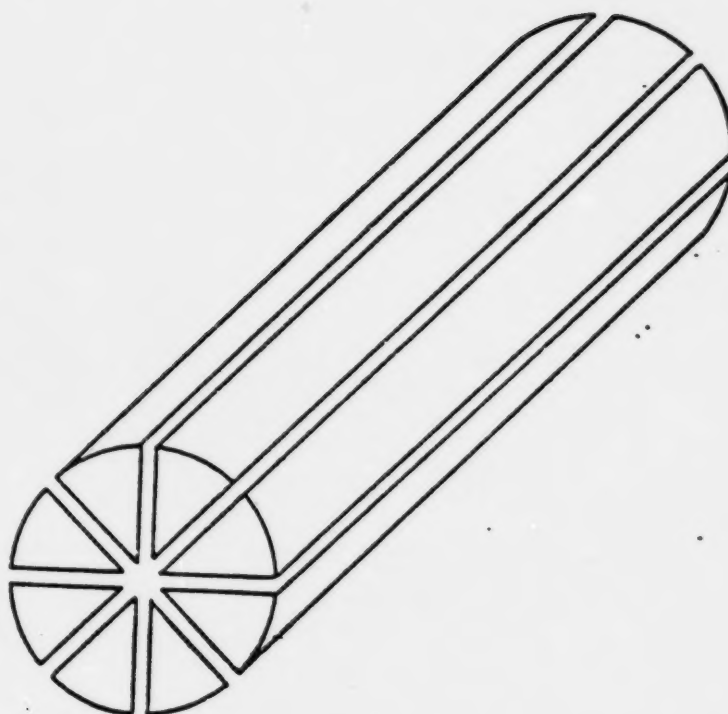


Figure 5.8 Internal view of conductivity cell: eight metal pieces are fixed around the circumference of the pipe to form star shaped electrode system

d) Conductivity meter: The conductivity meter unit (Figure 5.7) consists of a conductivity cell wired to a standard conductivity meter. The conductivity cell is connected 2.42 ft. downstream of the Magnetic flow meter. The main body of the conductivity cell is a cylindrical pipe made out of transparent acrylic. Eight metal pieces, equally spaced around the circumference, are fixed at the inside of the pipe so as to form a star-shaped electrode system. The internal view of the conductivity cell is shown in Figure 5.8.

5.4 Pressure Measurements (Fig. 5.9)

The pressure taps from each of the pipes and the differential pressure meters are all connected, upstream and downstream lines separately, to common manifolds (one for the upstream and one for the downstream) with stainless steel tubing. Because of the extreme range of flow rates involved, three glass manometers with different manometric fluids (chlorobenzene, density = 1.1 g/cc; carbon tetrachloride 1.595 g/cc; and Mercury 13.548 g/cc) are used for calibration. For the on-line metering, five validyne pressure transducers covering the whole range of pressure drops are installed. Their calibration are given in Fig. 5.10. The pressure transducer system and data acquisition system are shown in Fig. 5.15.

5.5 Calibration of Pipelines

Calibrations of the pipelines were necessary to check if they were hydraulically smooth and free of any burrs present at the inside of the pressure tap holes. To calibrate the pipelines, turbulent flow data were collected from each pipeline with water as the test fluid. This data was then compared with the Nikuradse equation for smooth pipes and fitted to within $\pm 2.5\%$.

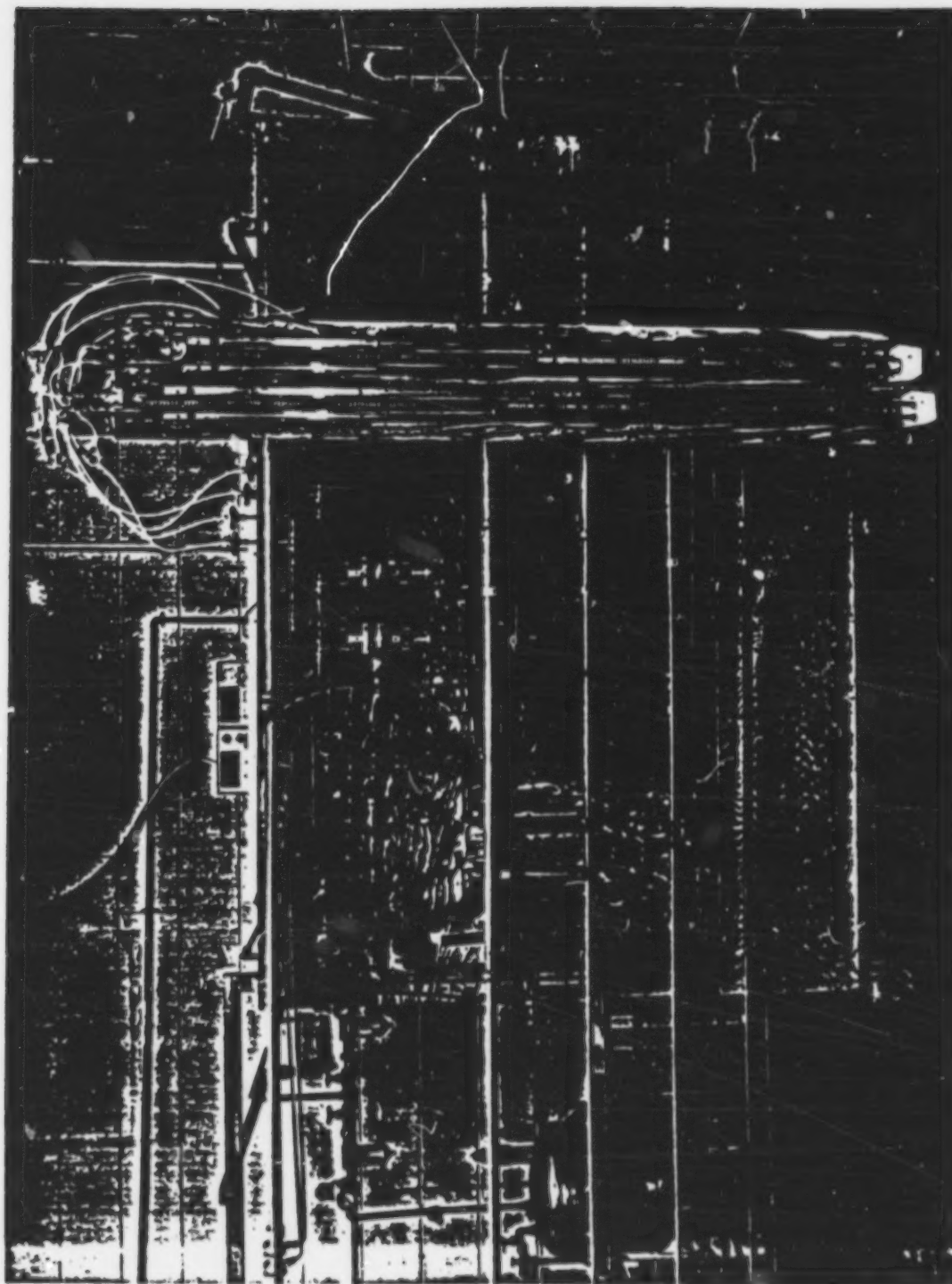


Figure 5.9: Pressure Measurement System

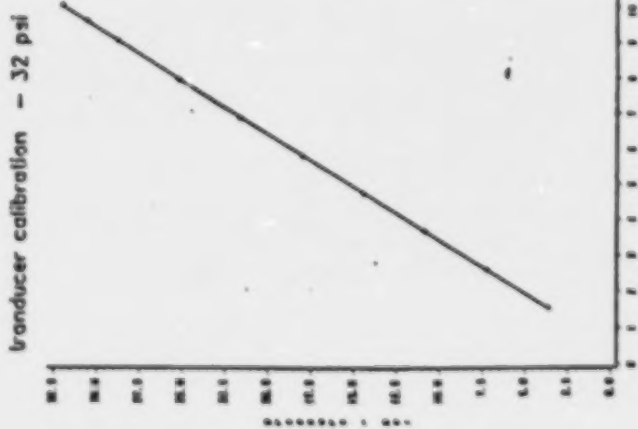
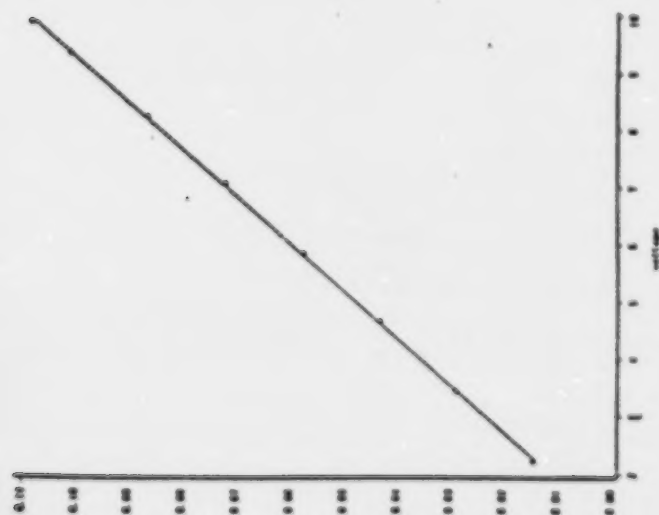
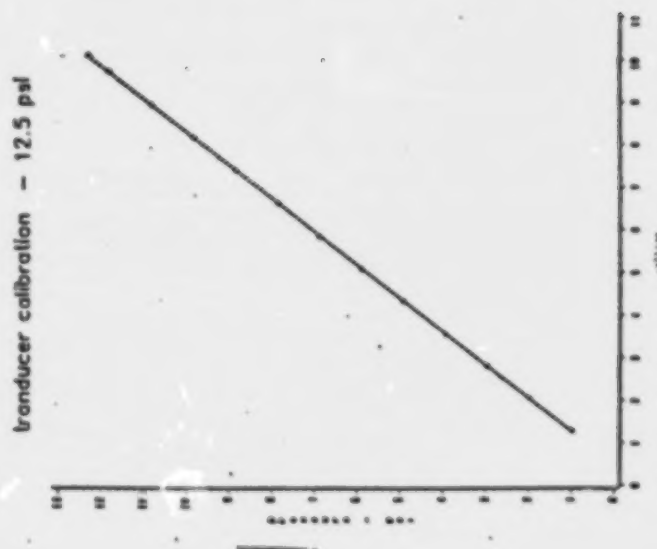
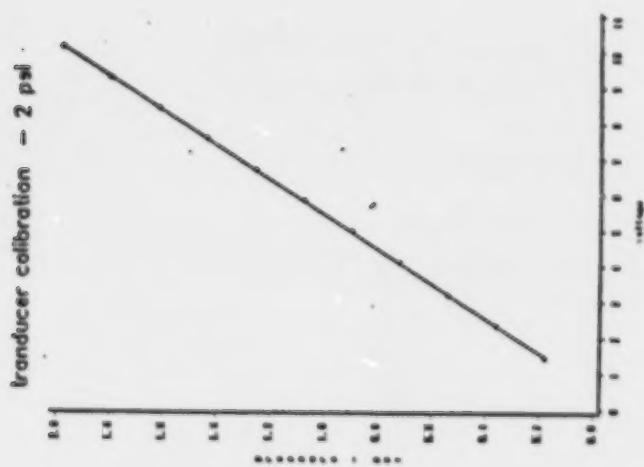
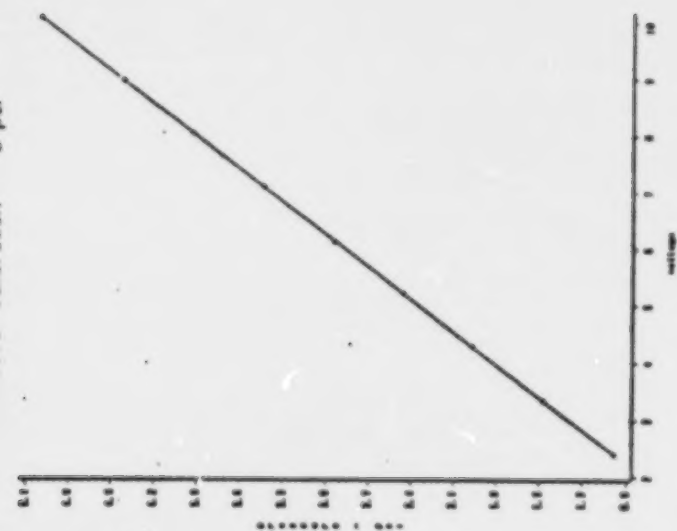


Figure 5.10: Pressure Transducer Calibrations

$$\frac{1}{\sqrt{f}} = 4.0 \log N_{Re} \sqrt{f} - 0.40 \quad (5.50)$$

5.6 Calibration of Orifice and Venturi meters

Before commencement of two phase experimental work, the orifice plate meter and venturi meter were calibrated with water. The calibration curves (C_D vs N_{Re}) were plotted for both the meters. (see Figs. 5.11 and 5.12) and found to be satisfactory.

$$C_D = \frac{W \sqrt{1 - \beta^4}}{A_0 \sqrt{2 \rho \Delta p}} \quad (5.60)$$

where

C_D = discharge coefficient

A_0 = area of orifice (or throat)

β = D_0/D , ratio of throat (or orifice) to pipe diameter

5.7 Calibration of Magnetic Flow Meter

A calibration curve for the magnetic flow meter is shown in Figure 5.13. Dial read out reading is plotted against the actual measured flow rate. There exists a linear relationship between the dial reading and flow rate.

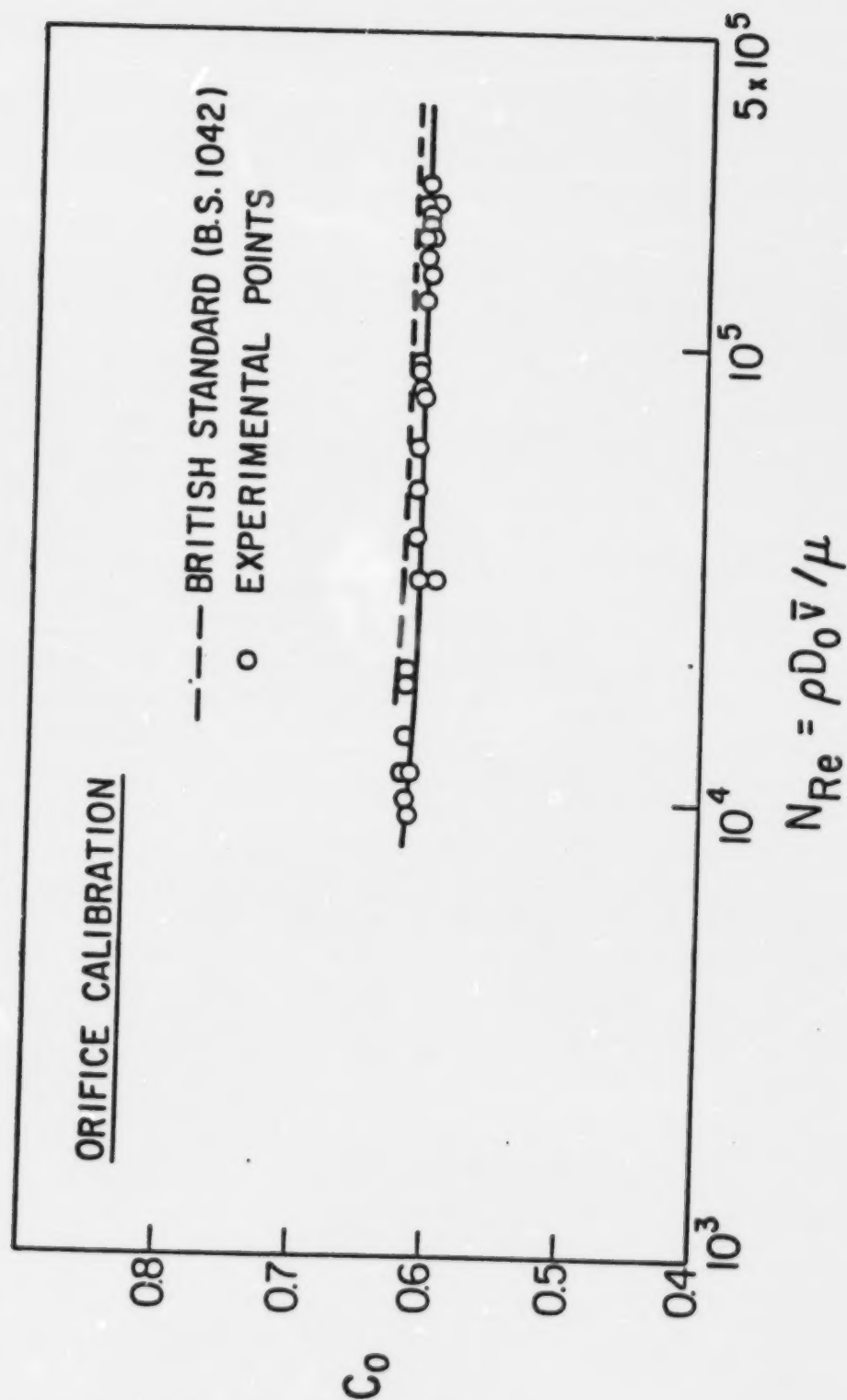


Figure 5.11: Orifice Coefficient Calibration

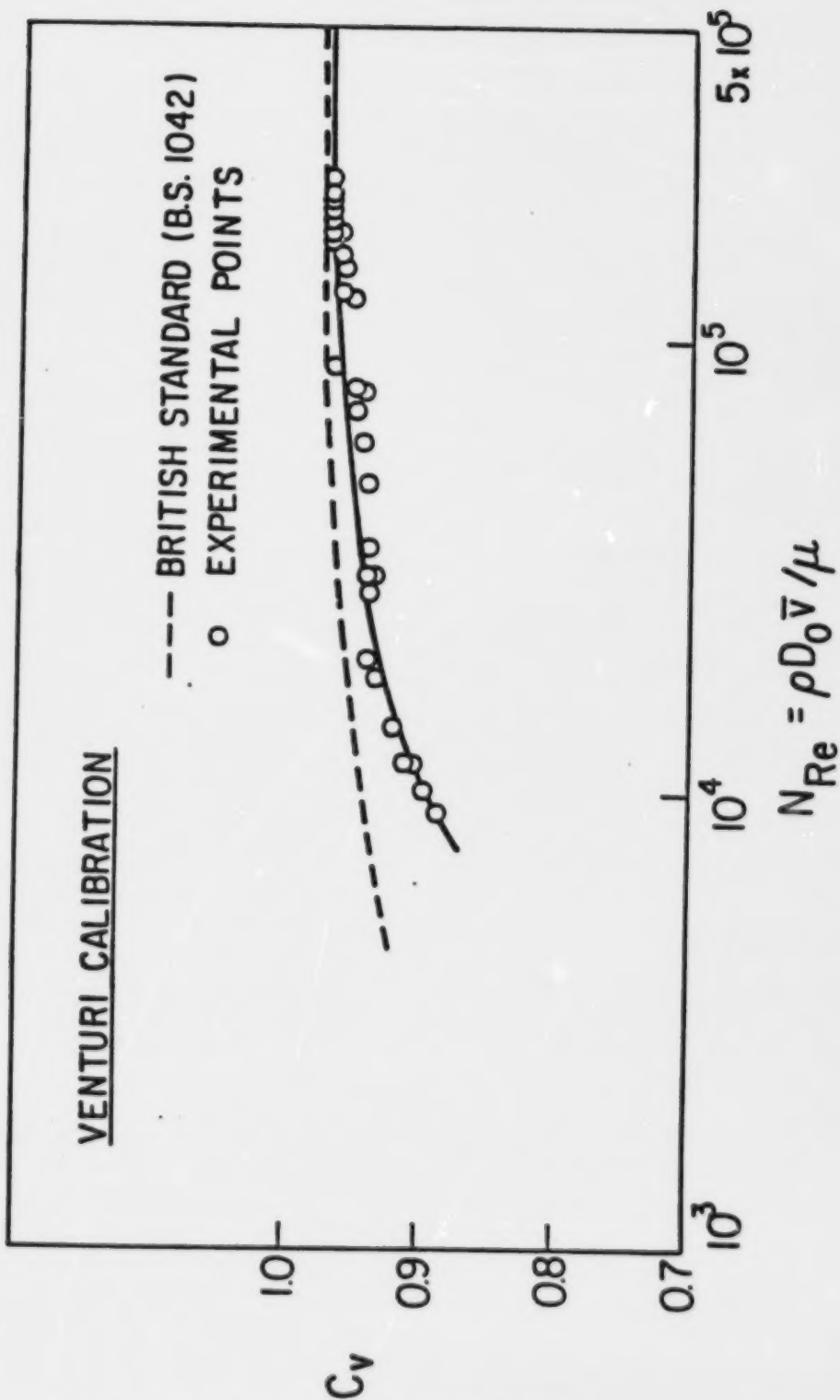


Figure 5.12: Venturi Coefficient Calibration

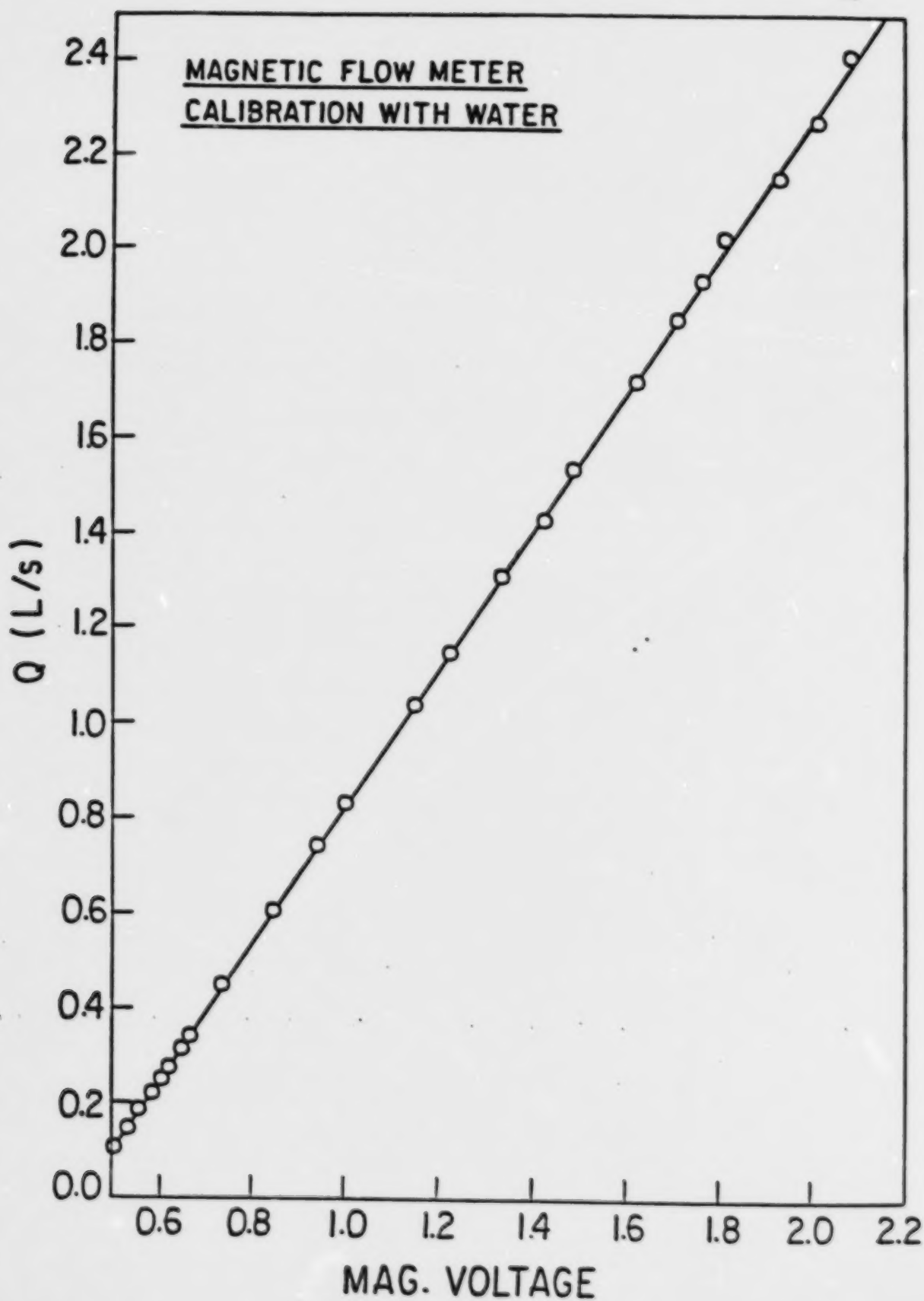


Figure 5.13

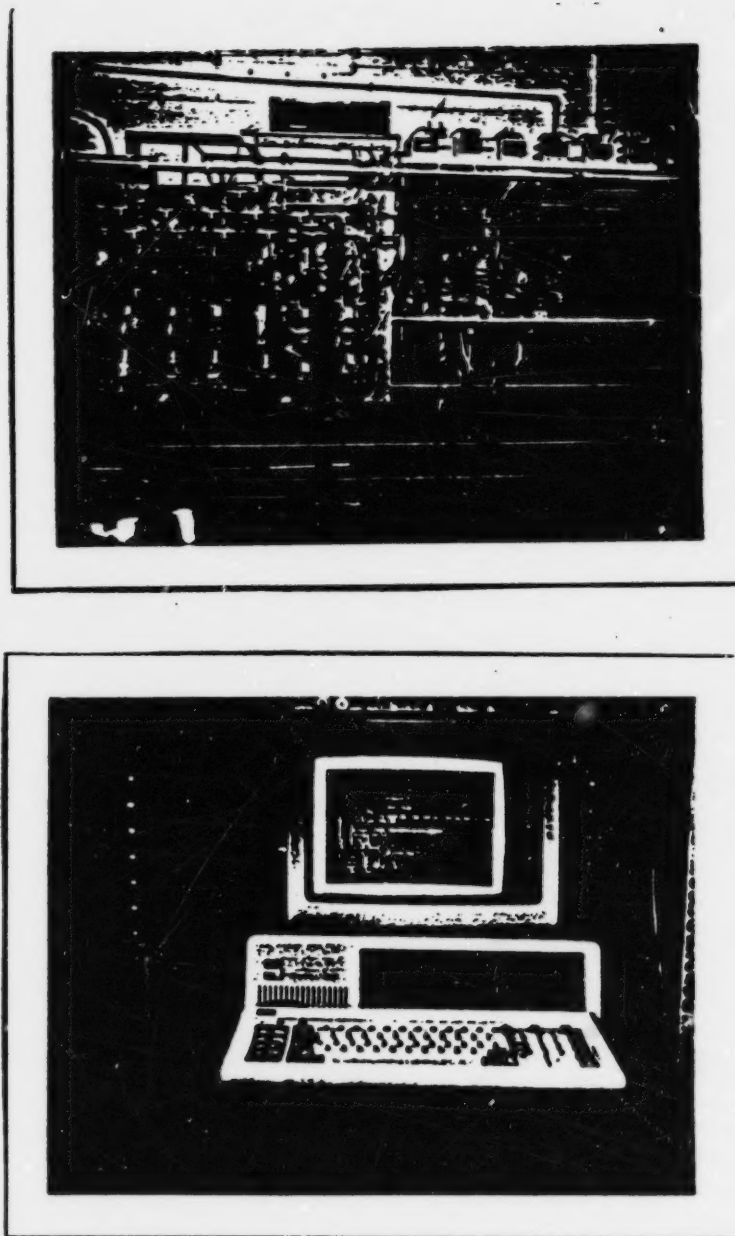


Figure 5.15

Pressure transducer system and computer data acquisition system

watch method) is entered into the computer and data from the metering instruments is collected automatically at the same time. The program allows sampling of all the labmaster channels which are connected to the metering instruments. From each channel, 250 data points are taken and the average of these 250 points is stored on the diskette as well as printed on the printer. In this way, measured flow rate versus instrument reading data are obtained.

Section B: High Flow Subroutine: After calibration of the instruments is done, the pressure drop data are collected from various pipelines. Now instead of measuring the flow rate manually, it is determined from the instruments already calibrated. Here again, the program allows sampling of channels connected to various instruments. From each channel, 250 data points are collected and the average of these 250 points is stored on the diskette as well as printed on the printer.

Section C: Low Flow Subroutine: In this section of the program, the laminar flow pressure drop data (i.e. at very low flow rates) is collected from the various pipelines. Since at very low flow rates, the flow metering instruments may not give accurate flow measurements, the flow rates have to be physically measured. This flow rate is then fed into the computer and the sampling of various pressure transducers and thermocouples is done. The data collected (250 points from each channel) is averaged and stored on the diskette as well as printed on the printer.

Section D: Plotting Subroutine: Software has been developed which allows the plotting of any of the recorded variables against any other variable. In addition, calculated quantities may also be automatically plotted.

Chapter 6

RHEOLOGICAL STUDIES OF EMULSIONS

The discussion of this chapter relates to
Tasks 1, 4 and 5 of the contract.

In Appendix 1, Papers 3a, 3b, 4, 5, 6a, 6b and 7 report in detail the work which has been done on the Rheology of various oil/water emulsions.

At the outset of the work, it had been envisaged that the Weissenberg Rheogoniometer (i.e. the disc and cone type) which was available in our laboratories would be used to investigate the Rheologies of the various emulsions. Paper 4, "Flow Behaviour of Oil in Water Emulsions", describes how, in preliminary work, a stable emulsion ranging in concentration from 40-80% oil in water, was systematically investigated using the Rheogoniometer. At this early stage it was felt that the Rheogoniometer was providing excellent results showing the effects of shear rate and oil concentrations on the viscosity. From this data, a concept of "maximum attainable concentration" was developed and introduced as a parameter in the relationship between relative viscosity and oil concentration to account for what appeared to be certain variations in the emulsion characteristics. The "maximum attainable concentration" appeared to be very closely related to the maximum concentration that could be experimentally achieved before inversion of the phases occurred. It was postulated that this method of correlating data (i.e. the introduction of the "maximum attainable concentration" as a parameter) would allow for the strong effect of the emulsifier concentration on

the Rheology. The emulsifier was shown to greatly effect the particle sizes of the emulsion. This observation was made by use of photomicrographs and microscopy. Paper 5 of Appendix 1 was a much shortened version of Paper 4 and it was presented at a gathering of distinguished Rheologists attending the IX International Congress of Rheology, Acapulco, Mexico on October 8, 1984. While the paper was well received, questions were asked about the validity of using the Rheogoniometer as a tool for measuring viscosity. In particular the question of slip effects at the walls of the instrument was raised.

Subsequently a much broader spectrum of experiments was undertaken (see Papers 3a and 3b of Appendix 1.) in which it was decided to investigate a variety of O/W and W/O emulsions in three different kinds of viscometers, namely; the Fann viscometer, the Weissenberg Rheogoniometer and the Brookfield viscometer. In both kinds of emulsion types (O/W and W/O) it was found that the data obtained above certain limiting dispersed phase concentrations (70% for O/W, 60% for W/O) were different depending on the type of apparatus being used. In fact the viscosity measurements of the various instruments was significantly different. This variation in the flow curves for the same concentration of emulsions was attributed to the presence of "slip effects", especially in the cone and plate viscometer (Rheogoniometer) which had a large surface area and very narrow gap width. Further evidence of the slip effect being a factor in the Rheogoniometer results was that the emulsions exhibited no yield stress while the other two instruments indicated a definite yield stress effect. The question of possible particle breakdown in the Rheogoniometer was shown to be not a factor.

This result was obtained from careful visual (microscopic) observation of the emulsions before and after testing.

As a result of this broad spectrum of experimental work it was concluded that the Rheogoniometer was not a suitable instrument for measuring the true characteristic viscosity of an emulsion. And from that time on, the Rheogoniometer was abandoned as a measuring instrument.

In Papers 6a and 6b of Appendix 1 the viscosity data obtained for six systems of both O/W and W/O kinds were obtained using only the Brookfield and Fann viscometers. Effects on viscosity such as the concentration of emulsifier, speed and time of mixing of the emulsion, and age of emulsion were all investigated.

A new method of correlation of viscosity data based on a parameter $\nu_r=100$ which is the concentration at which the viscosity of the emulsion reaches one hundred times the viscosity of the continuous phase. It was argued that this parameter will take into account the effects of all the variables on a given emulsion species. The proposed correlation was:

$$\nu_r = \left[1 + \frac{1.342 \phi / \phi_{\nu_r=100}}{1.194 - \phi / \phi_{\nu_r=100}} \right]^{2.226} \quad (6.1)$$

This correlation is very useful because it will allow the predictions of a complete viscosity/concentration relationship from one viscosity measurement at one finite concentration. The correlation was successfully tested as a method for predicting the concentration/viscosity relationship of some Cold Lake emulsions.

well-known Blasius equation:

$$f = 0.079/(N_{Re})^{0.25} \quad \dots 3.37$$

becomes after substitution of the expressions for f and N_{Re} :

$$\frac{D\Delta p}{4L} = 0.079 / \left(\frac{\rho \bar{V} D}{\mu} \right)^{0.25} \quad (3.37)$$

or

$$\frac{D\Delta p}{4L} = \frac{0.079(\rho)^{0.75}(\mu)^{0.25}(D)^{1.5}}{2(8)^{1.75}} \left[\frac{8\bar{V}}{D} \right]^{1.75} \quad (3.38)$$

which can also be written as:

$$\frac{D^{1.25}\Delta p}{4L} = 0.079 \frac{(\rho)^{0.75}(\mu)^{0.25}}{2} [\bar{V}] \quad (7.1)$$

Thus for Newtonian fluids, if we plot $\ln(D\Delta p/4L)$ versus $\ln(8\bar{V}/D)$ according to equation (3.38) for data obtained in a variety of pipeline diameters, we should produce a family of straight-line curves having the slope of 1.75. On the other hand, if we plot $\ln(\Delta p D^{1.25}/4L)$ versus $\ln(\bar{V})$ according to equation (7.1) for different diameter pipelines, we should obtain a single straight line with a slope of 1.75 and an intercept of $[0.079(\rho)^{0.75}(\mu)^{0.25}]$. From this

intercept, and knowing the density of the fluid, we should be able to calculate the viscosity of the fluid.

As the shear rates in turbulent flow are very high, many non-Newtonian fluids are known to behave similarly to Newtonians in that they exhibit a relatively constant viscosity when it is derived in the above manner. The viscosity thus derived is generally referred to as the effective turbulent viscosity μ_{eff} and equations (3.37) (3.38) and (7.1) work well when μ_{eff} is used in this way. In treating the viscosity as described, we are implying that the emulsion, which is a fine dispersion, can be treated as a single phase fluid. Emulsions are known to be pseudoplastic, i.e. their viscosity decreases with the shear rate and at very high shear rates the viscosity becomes constant. It is therefore implied that, at the very high shear rates experienced in the highly turbulent flow, the fluids are behaving like Newtonian fluids at the limiting (high shear) pseudoplastic viscosity.

7.3 Results and Discussion

Figures 7.1-7.7 show the plot of $\Delta p D/4L$ versus $8\bar{V}/D$ on a log-log scale for various concentrations of emulsions. For each of the emulsion, we have obtained a family of straight parallel curves having a slope of approximately 1.75. This indicates that these fluids are behaving like Newtonian fluids (at least in turbulent flow).

To determine the effective turbulent viscosity for each of the emulsions, the data was replotted as $(\Delta p)D^{1.25}/4L$ versus $\bar{V}$ on a log-log plot. Figures 7.8-7.14 show these plots. The data for all the different pipelines for each emulsion fall on a single curve having a slope of approximately 1.75. The intercept of these

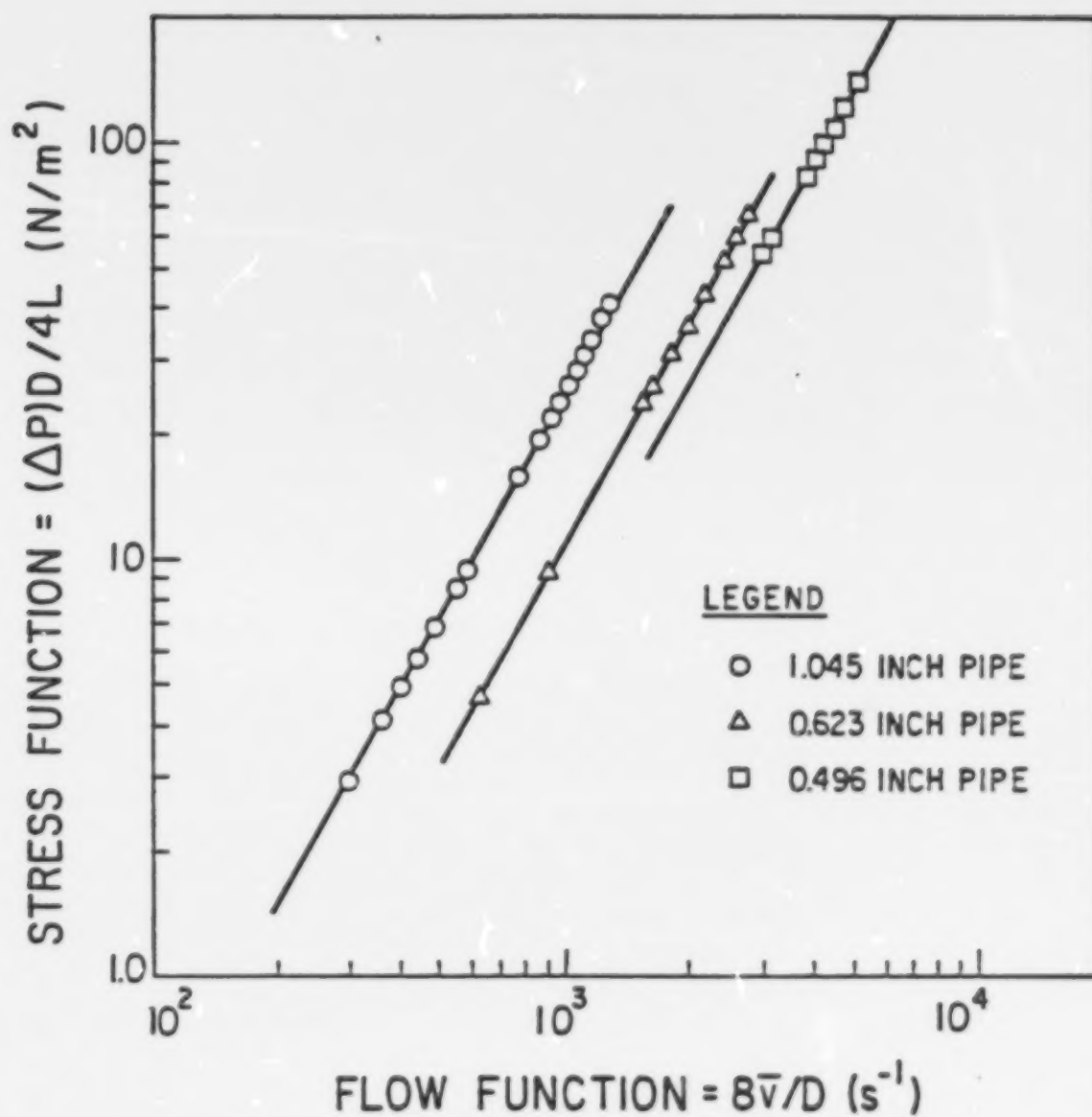


Fig. 7.1 Stress-Flow Diagram for Water

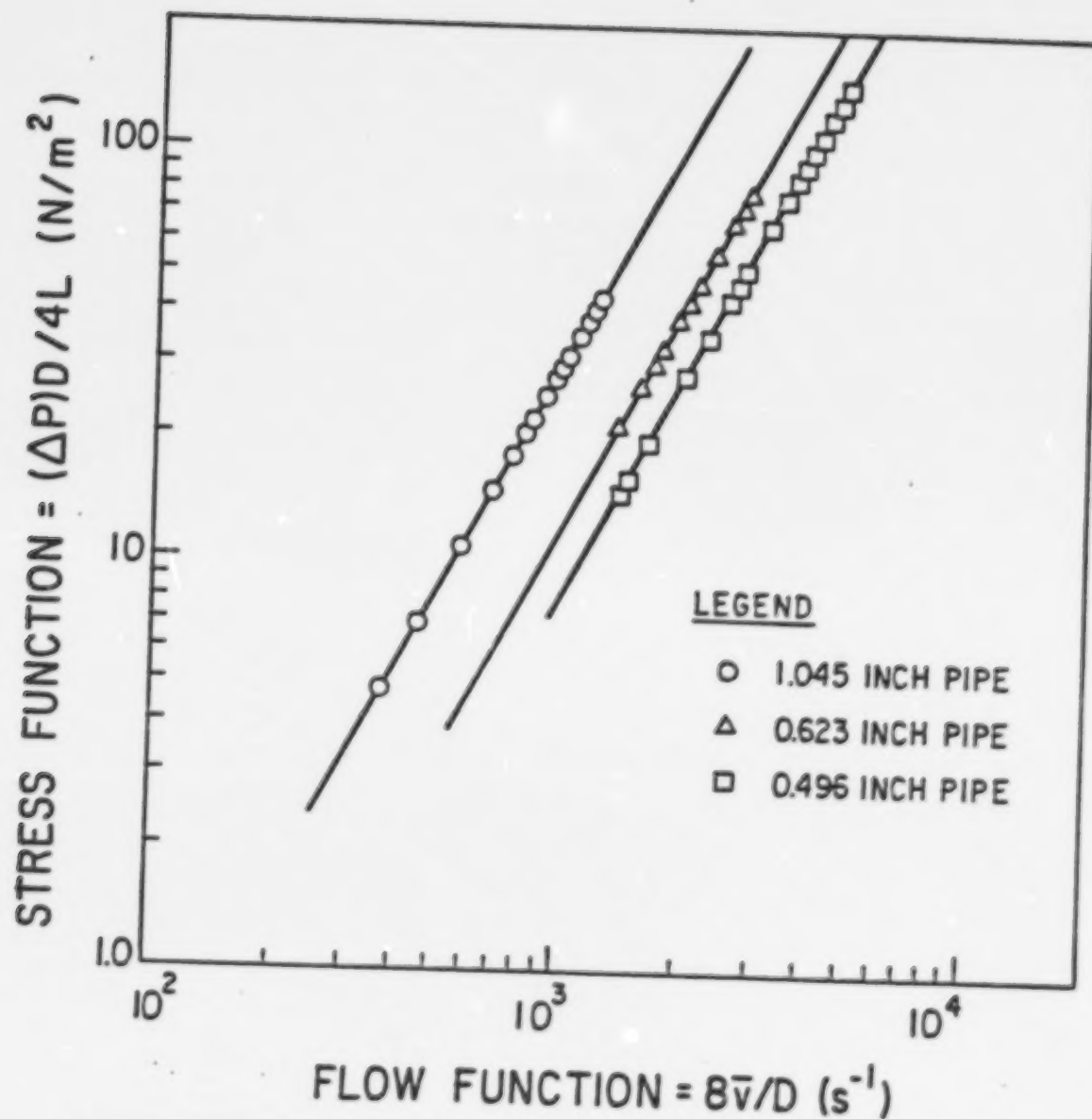


Fig. 7.2 Stress-Flow Diagram for 22.9 Percent Oil-in-Water Emulsion

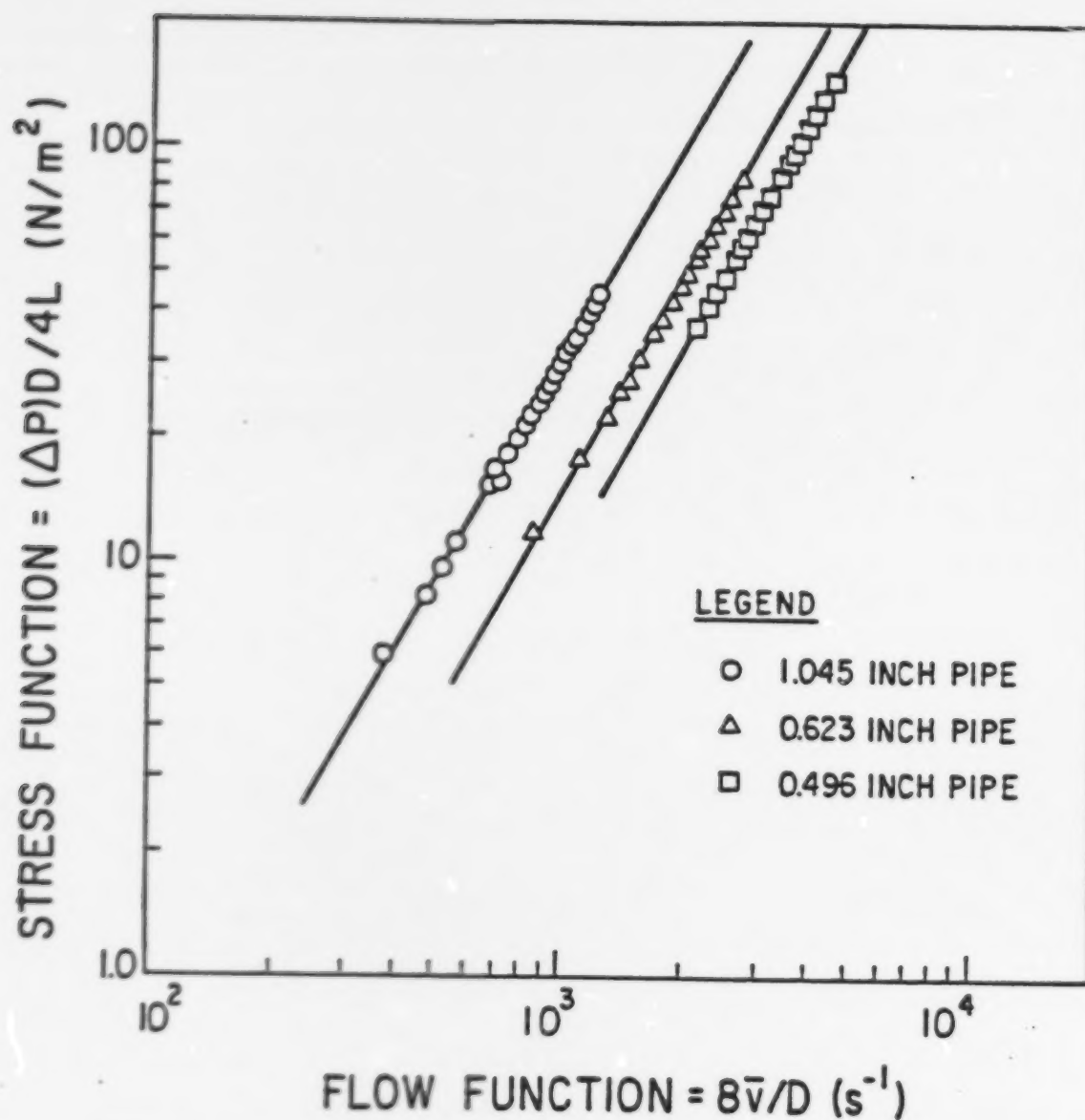


Fig. 7.3 Stress-Flow Diagram for 41.2 Percent Oil-in-Water Emulsion

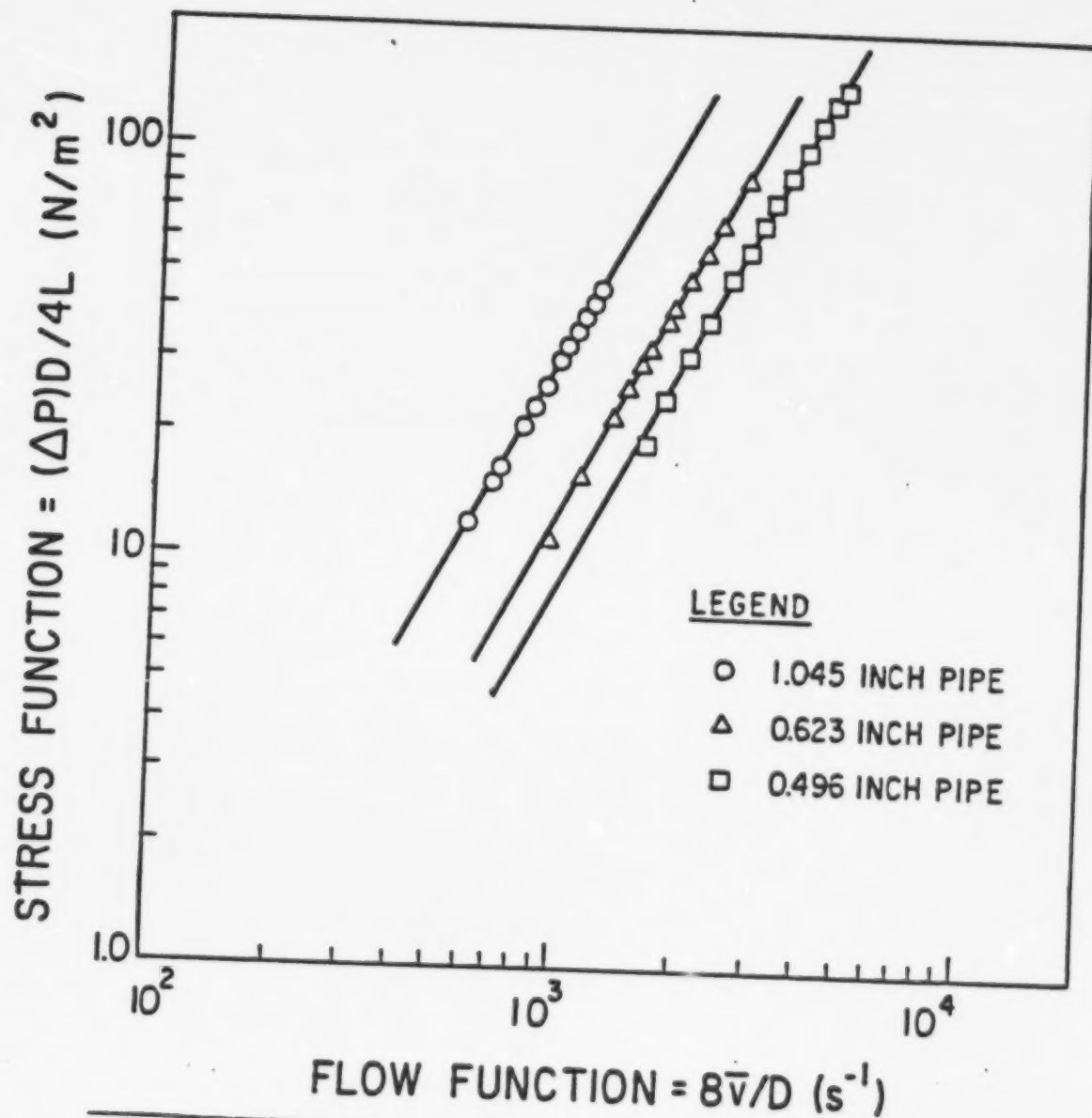


Fig. 7.4 Stress-Flow Diagram for 49.5 Percent Oil-in-Water Emulsion

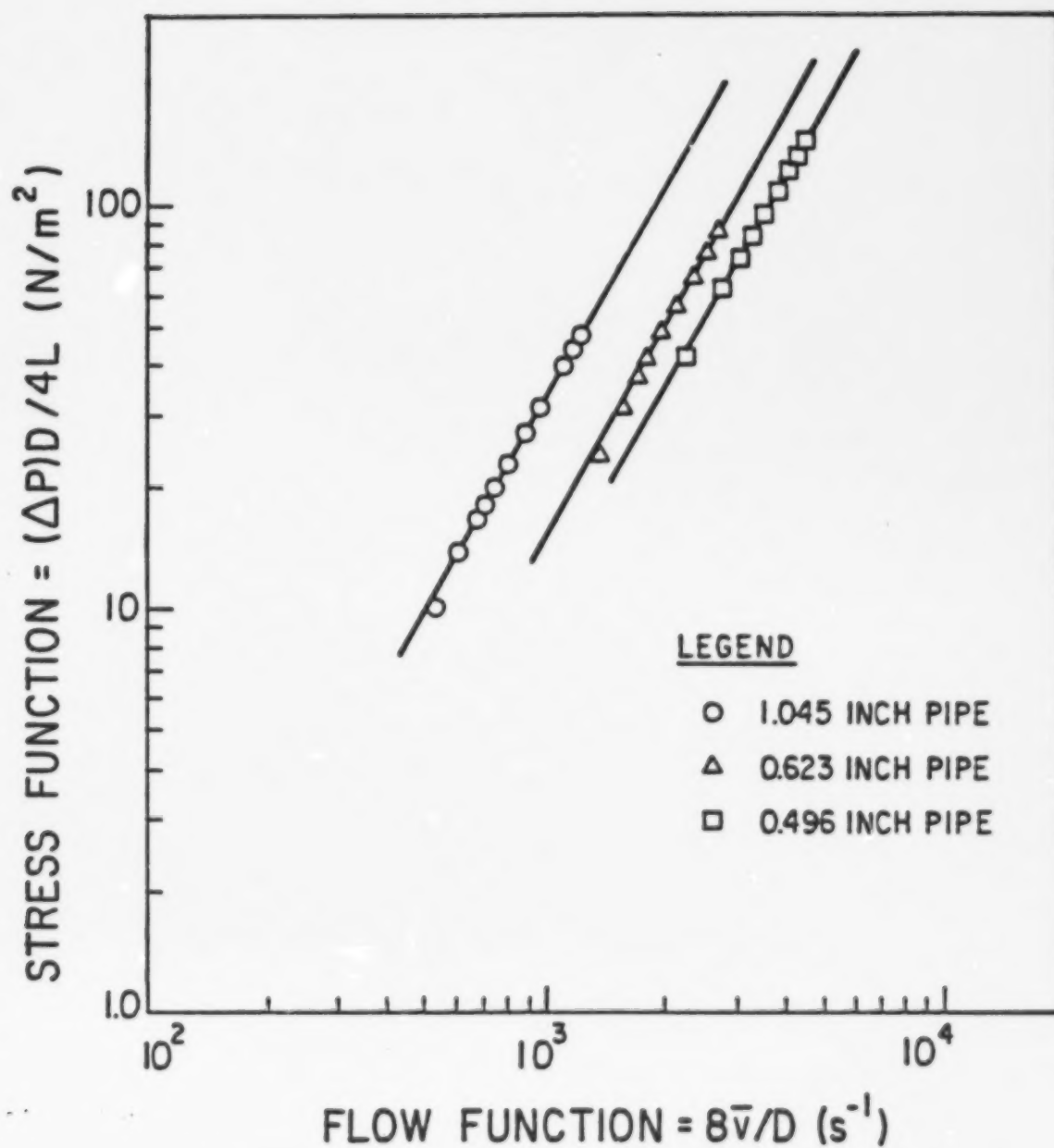


Fig. 7.5 Stress-Flow Diagram for 55.86 Percent Oil-in-Water Emulsion

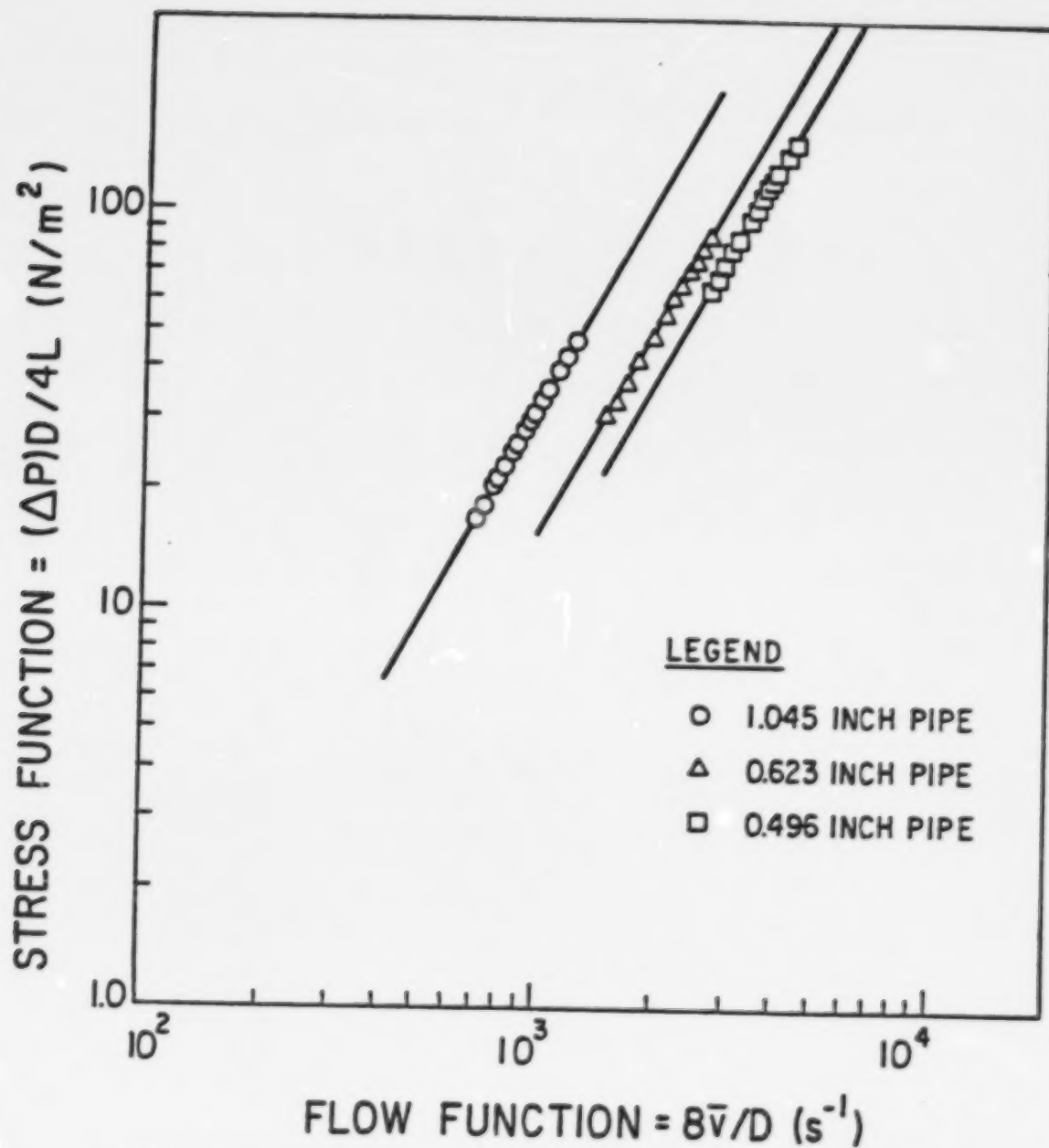


Fig. 7.6 Stress-Flow Diagram for 60.66 Percent Oil-in-Water Emulsion

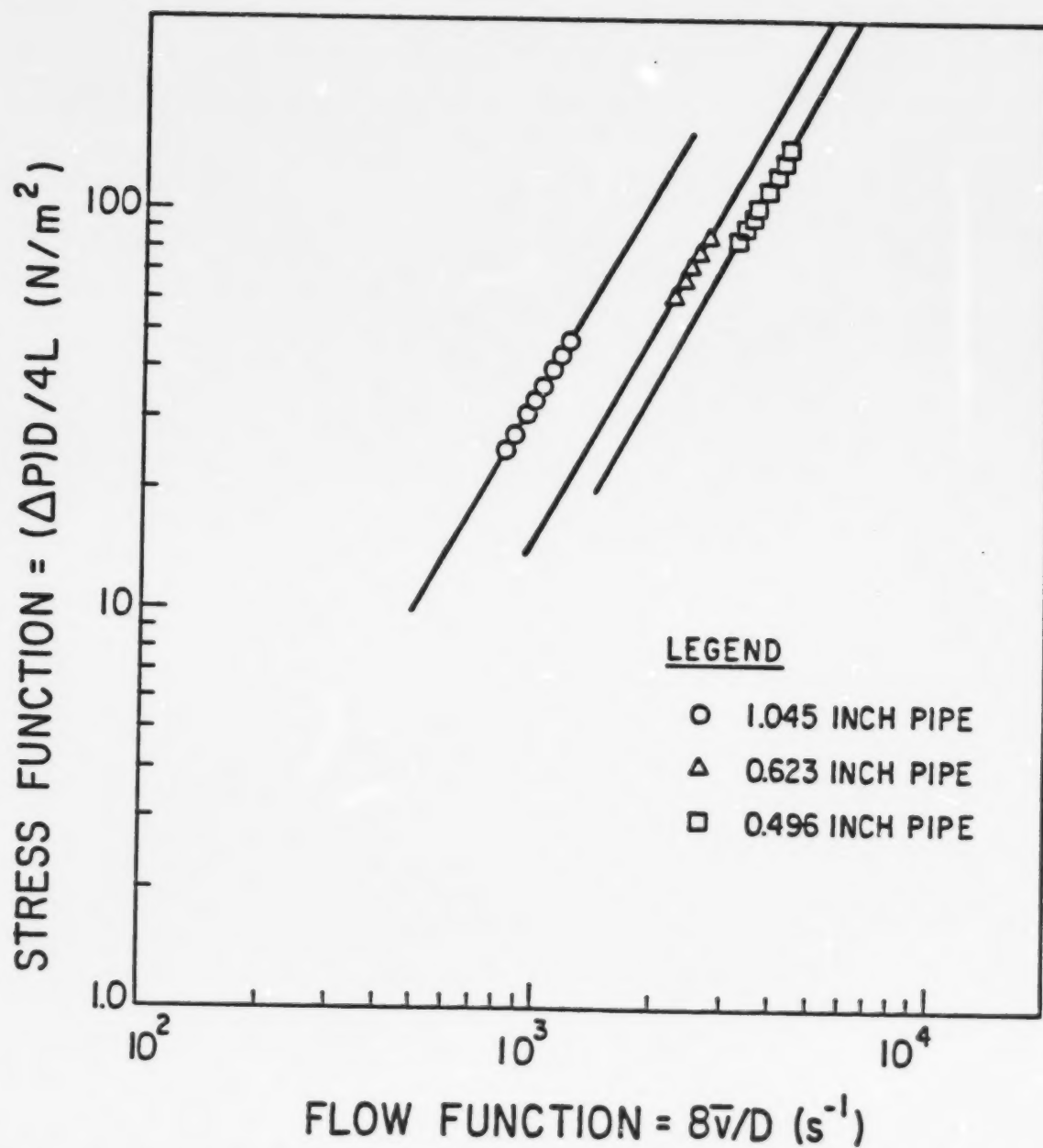


Fig. 7.7 Stress-Flow Diagram for 63.76 Percent Oil-in-Water Emulsion

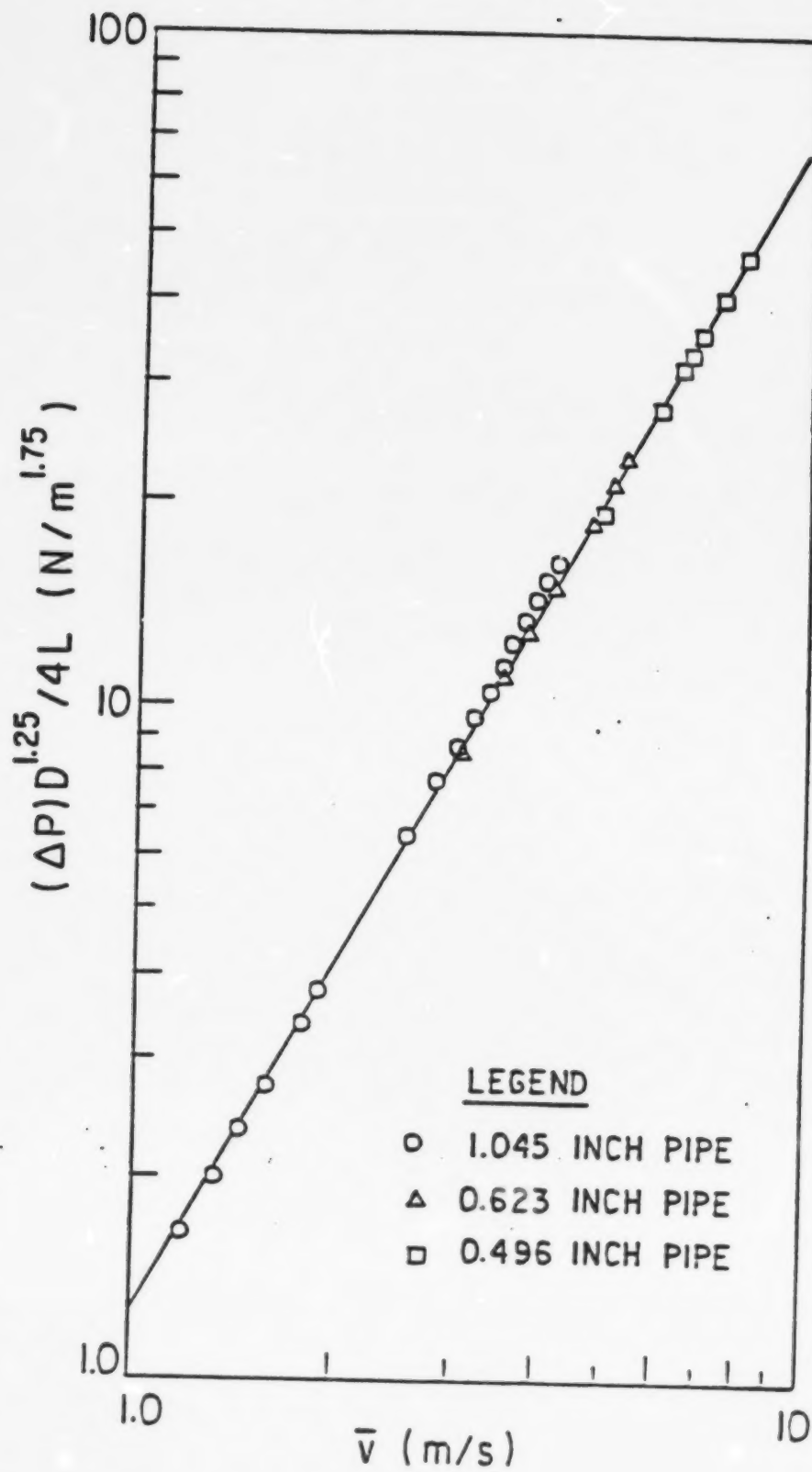


Figure 7.8: Data for Water

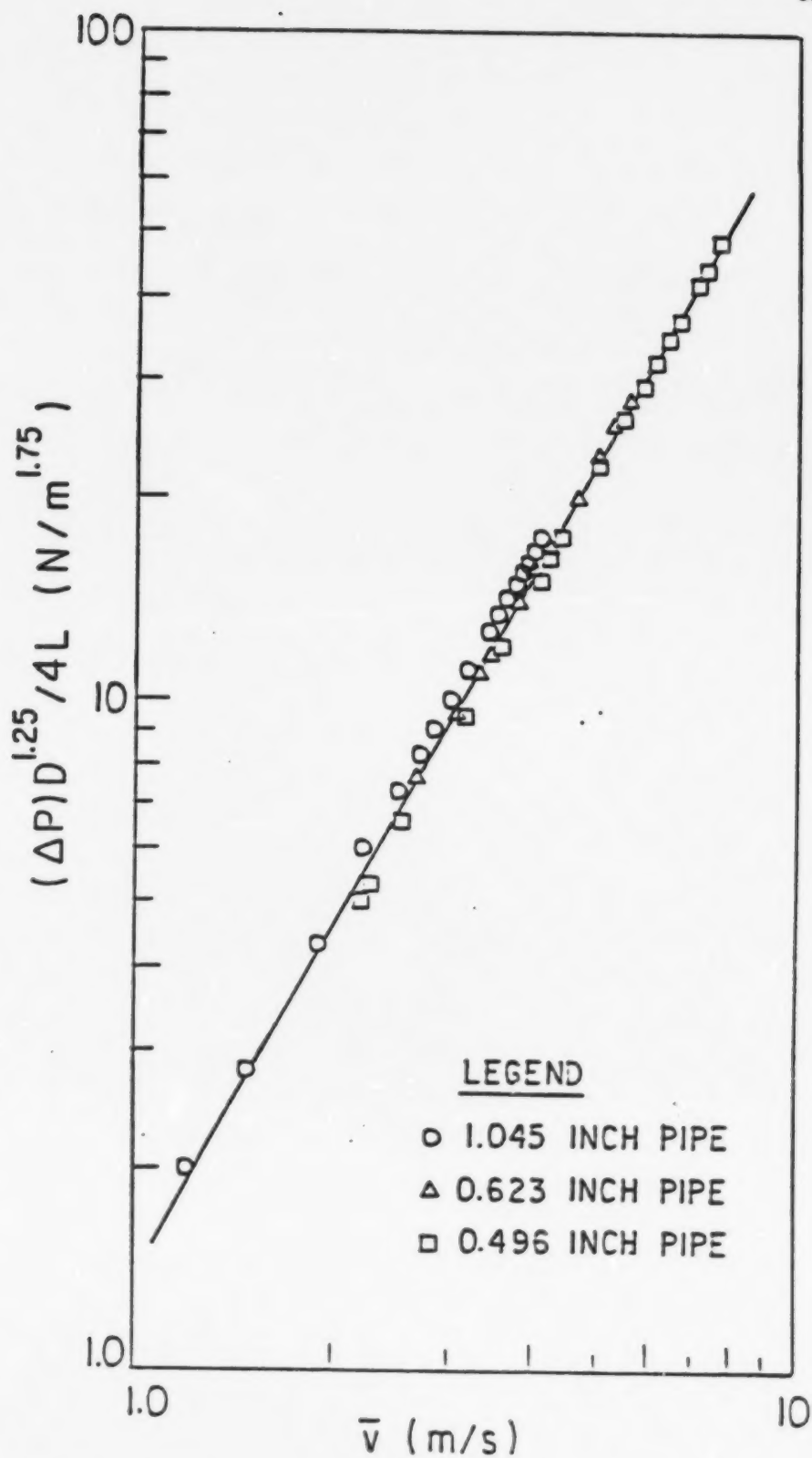


Figure 7.9: Data for 22.9 Percent Oil-in-Water Emulsion

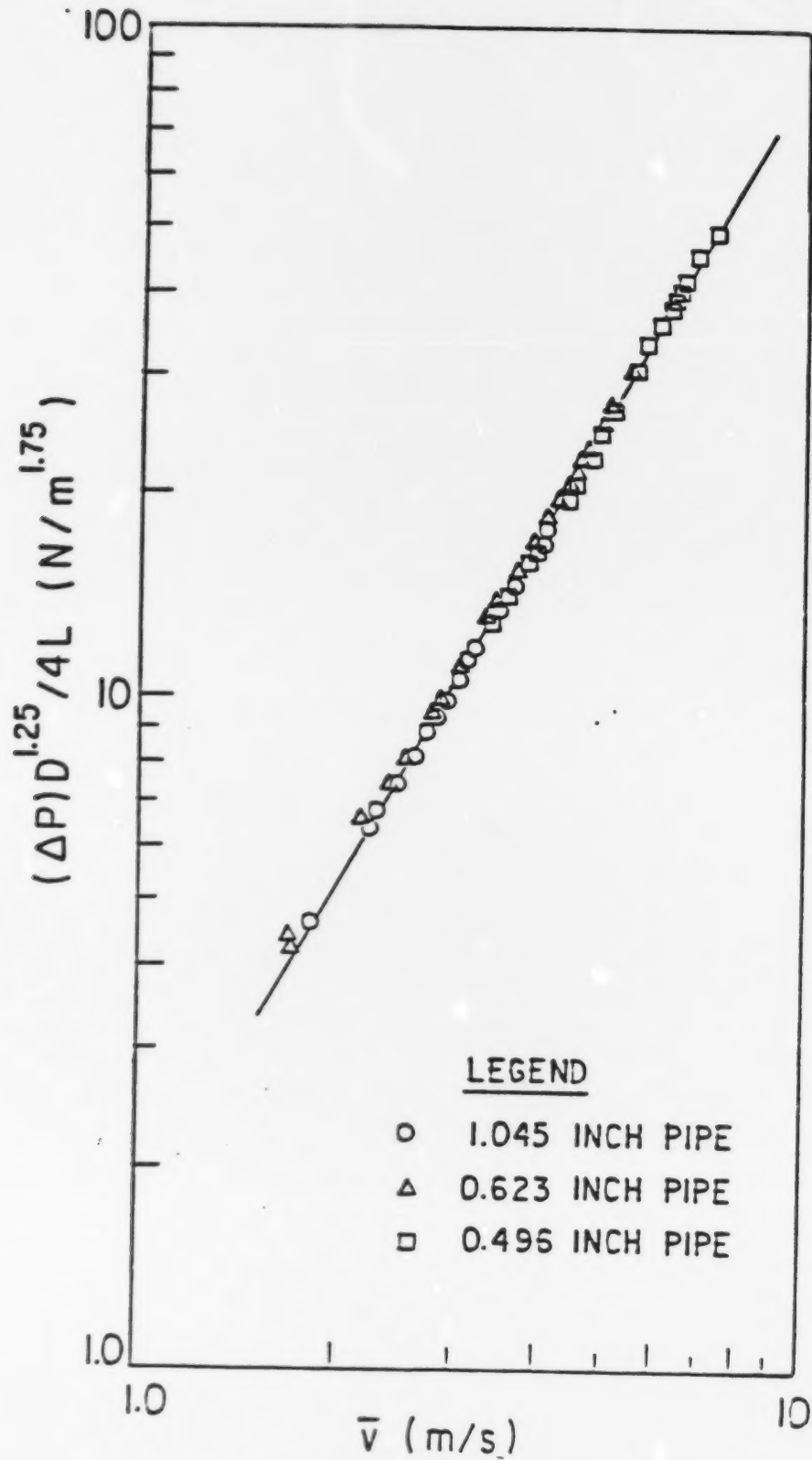


Figure 7.10: Data for 41.2 Percent Oil-In-Water Emulsion

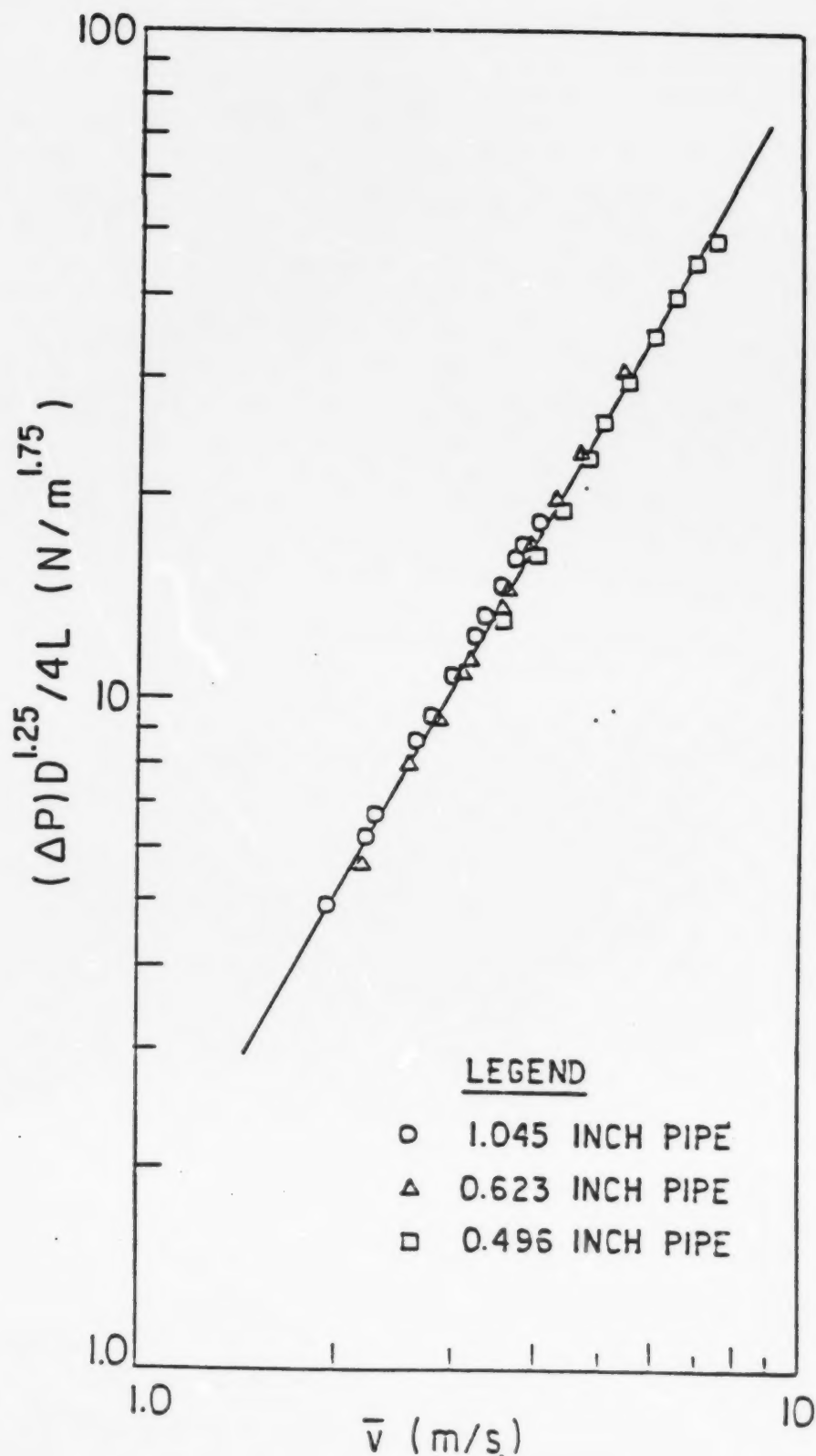


Figure 7.11: Data for 49.5 Percent Oil-In-Water Emulsion

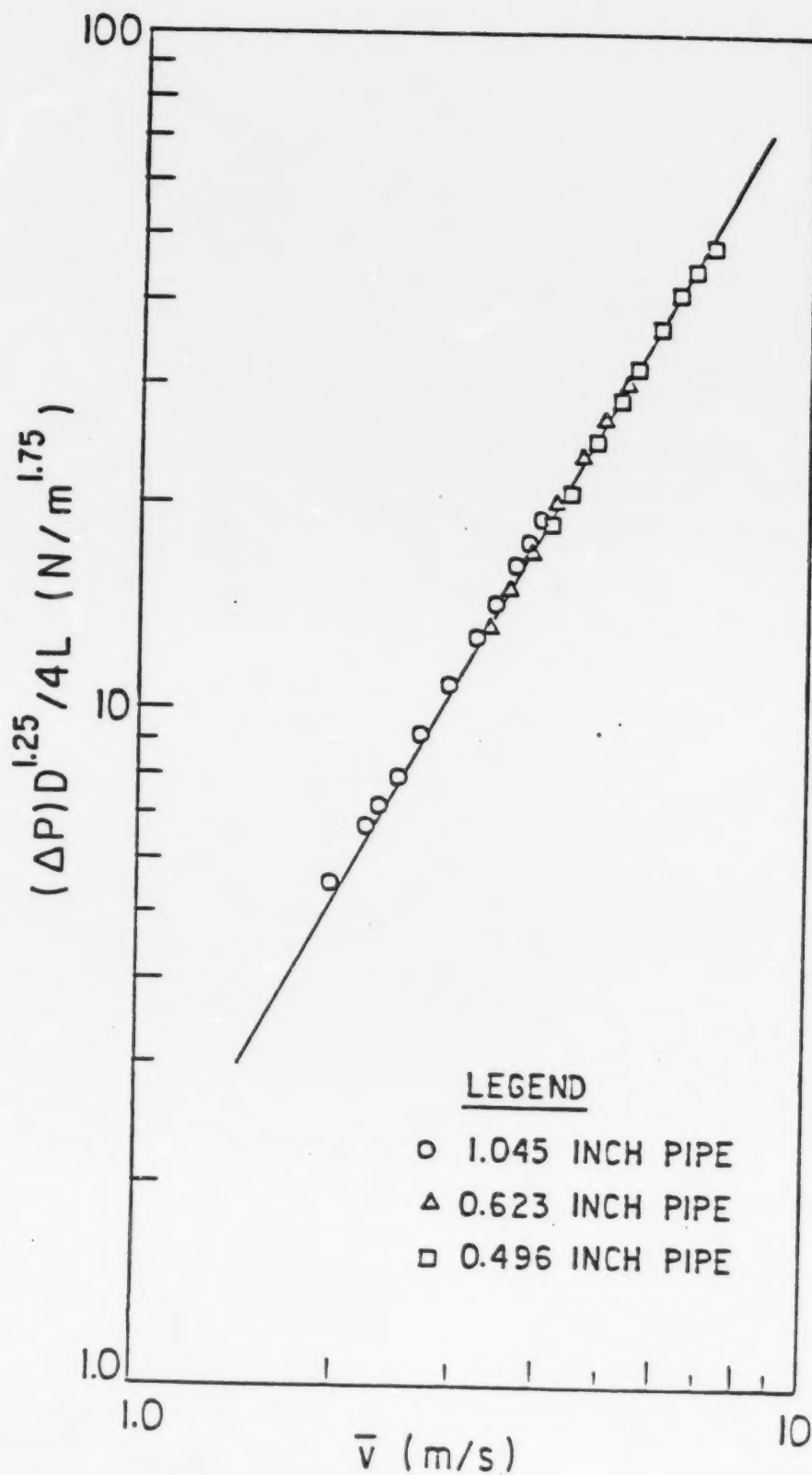


Figure 7.12: Data for 55.68 Percent Oil-In-Water Emulsion

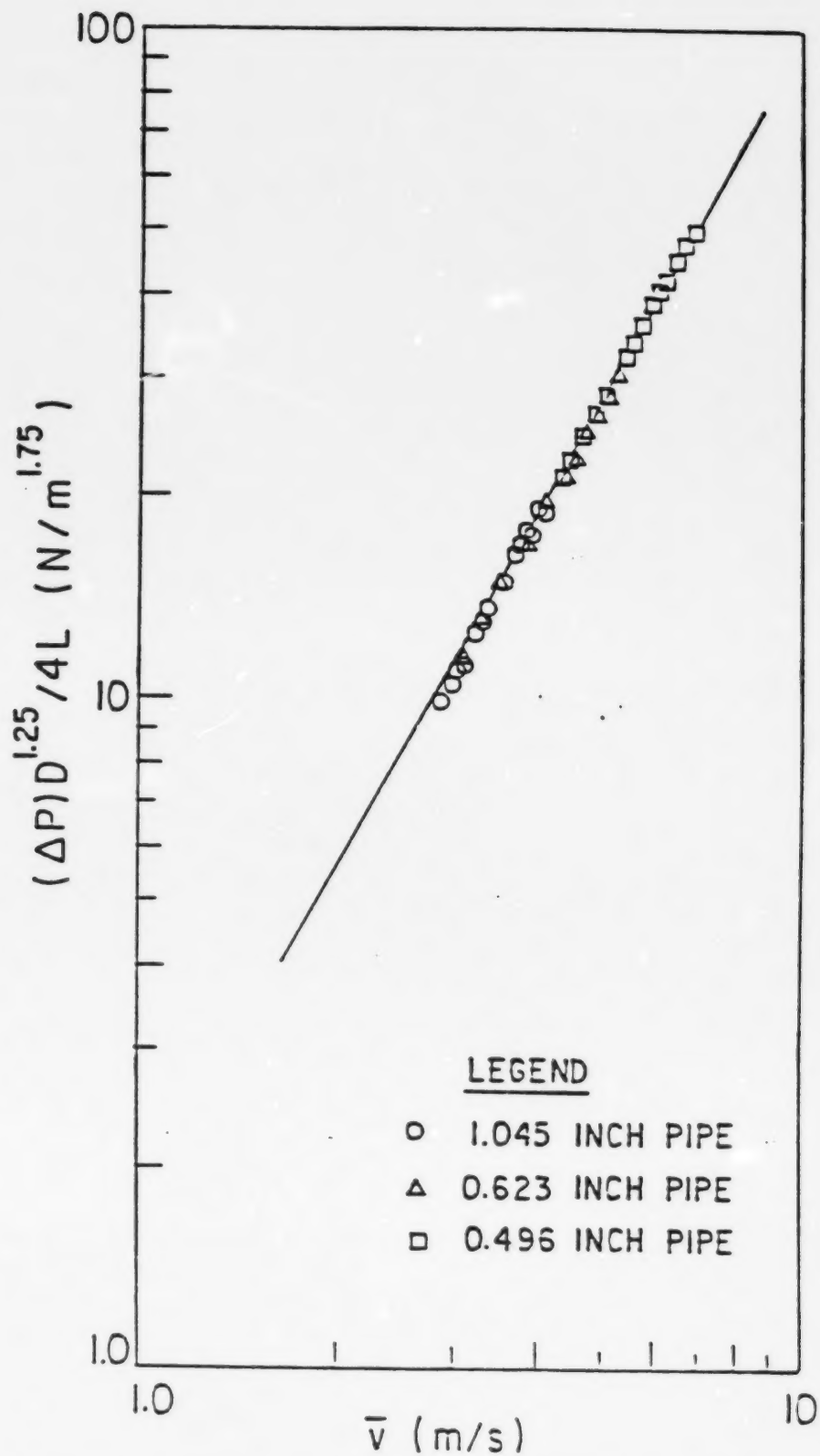
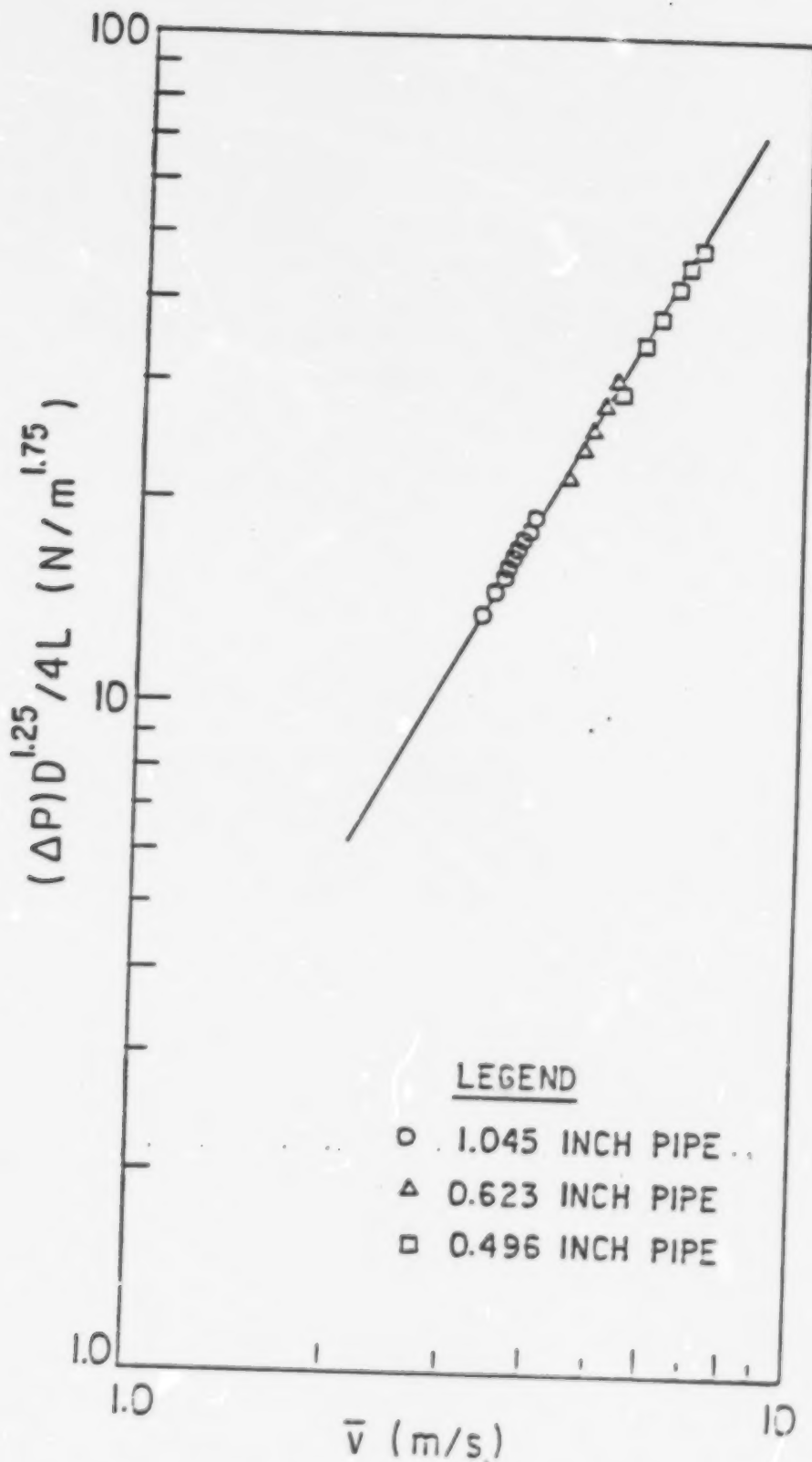


Figure 7.13: Data for 60.66 Percent Oil-In-Water Emulsion



curves gives the effective turbulent viscosity.

Figure 7.15 shows the variation of $\nu_r (= \nu_{eff}/\nu_{water})$ with the oil concentration. The relative viscosities are found to be lower than Vand's equation and much higher than Einstein's equation (see Paper 6b Appendix 1 for literature review). The following equation was found to be the best fit for our data:

$$\nu_r = \frac{\nu_{eff}}{\nu_{water}} = \exp (2.7\phi) \quad (7.2)$$

This follows the general form of Richardson.

Now, since we know that unstable oil-in-water emulsions are behaving like Newtonian fluids in turbulent flow, we can use the Blasius expression (Eqn. 3.37):

$$f = \frac{0.079}{(\frac{\rho D \bar{V}}{\nu_{water}})^{0.25}} \exp (0.675 \phi) \quad (7.3)$$

or

$$f = f_0 \exp (0.675 \phi) \quad (7.4)$$

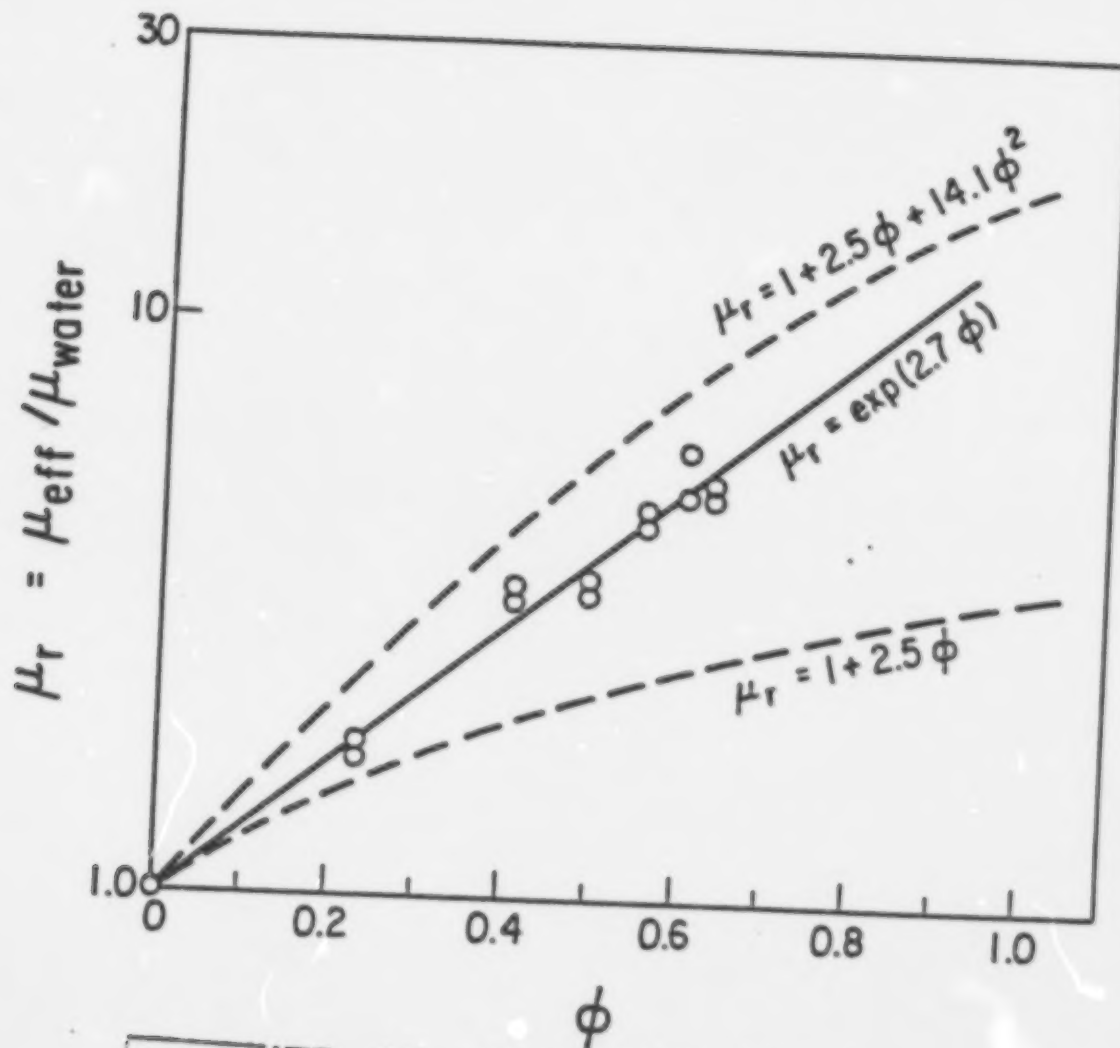


Fig. 7.15 Relative Turbulent Viscosity versus the Oil Concentration

Where f_0 is the friction factor when only water is flowing at the same mass flow rate as the emulsion in the same diameter pipe.

Figure (7.16) shows the plot of $f_{\text{predicted}}$ from equation (7.4) versus f_{measured} ($\Delta p/4L$, determined from the pressure drop measurements). The agreement between $f_{\text{predicted}}$ and f_{measured} is found to be quite good with uniform scatter for the various different emulsions.

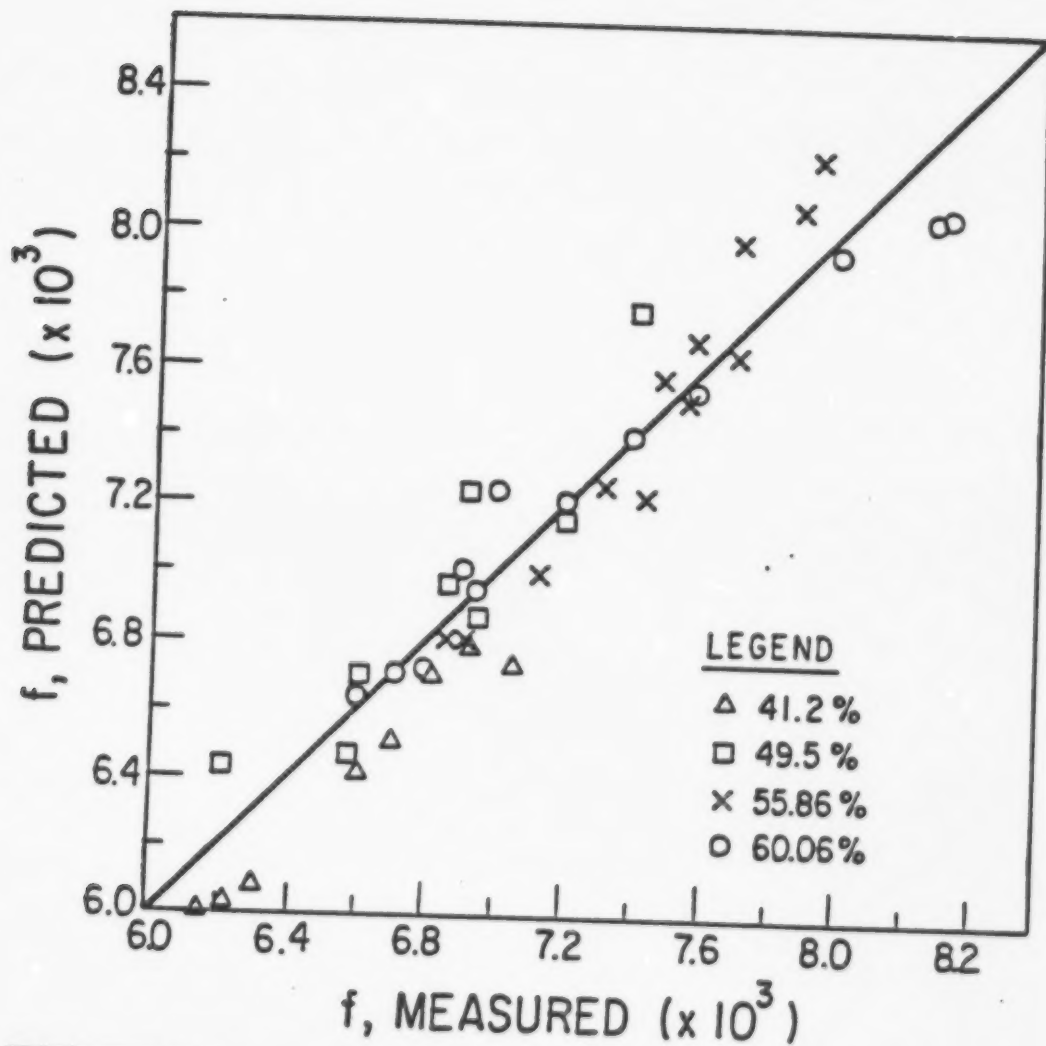


Fig. 7.16 Friction Factor Predicted from Correlation versus Measured One

Chapter 8

FLOW MEASUREMENTS OF EMULSIONS

The experimental results given in this Chapter relate to
Tasks 1, 2, 3, 5, 6 and 7

8.1 Experimental

As described above in Chapter 7, a further complete set of data was obtained for the flow of Bayol-35 and water in the various elements of the flow metering section of the oil/water flow loop. The total flow measurements were checked by use of the very accurate stop watch - measuring tank method. The oil holdup was pre-arranged by mixing known quantities of oil and water in the mixing tank.

8.2 Theoretical8.21 Orifice and Venturi Meters

The mass flow rate of a single phase fluid through a differential pressure meter (such as an orifice or a venturi) is given by:

$$W = \frac{C_D A_0}{\sqrt{1-\beta^4}} \sqrt{2\rho \Delta p} \quad (4.20)$$

If we assume that an emulsion will behave like a single fluid, we should be able to use equation (4.20) by incorporating an average density ρ which is weighted according to the oil hold-up. Thus;

$$W = \frac{C_D A_0}{\sqrt{1-\beta^4}} \sqrt{2\rho \Delta p} \quad (8.20)$$

or

$$\ln W = \ln \left(\frac{C_D A_0}{\sqrt{1-\beta^4}} \right) + \frac{1}{2} \ln (\rho \Delta p) \quad (8.21)$$

For high Reynolds numbers in single phase flow, C_D has a constant value (approximately 0.61 for the orifice and 0.97 for the venturi). At lower flow rates, C_D is known to vary with Reynolds number. If we assume that the value of C_D when the emulsion is flowing is the same as the value when the continuous phase is flow at the same mass flow rate, then we will obtain a single curve of W versus $\rho a p$ on the log-log graph for both the emulsion and the single continuous phase. In the flow rate region where C_D is constant, the curve will be a straight line.

8.22 Magnetic Flowmeter

The basic principle of the magnetic flowmeter is that an e.m.f. is induced in a fluid flowing normally to the magnetic fluid. The magnitude of the induced e.m.f. is proportional to the flow rate. The meter consists of field coils placed round the pipe to produce the magnetic field. Two electrode probes, inserted inside, pick up the induced e.m.f. which can then be read on a voltmeter after amplification. The calibration of the instrument is independent of the properties of the fluid except that it should be electrically conductive. This instrument gives the total volumetric flow rate of the flowing fluid.

8.23 Conductance Probe

The conductance probe continuously measures the conductance of the flowing emulsion. Since the conductivity of water is usually very high as compared to that of oil, a small change in volume fraction will give a measurable change in the signal. This technique is more useful for O/W emulsions where conductive water forms the continuous phase. For pure W/O emulsions, this technique does not work because

non-conductive oil phase forms the continuous phase.

The conductivity of O/W emulsions is related to the volume fraction of the oil by the following equation

$$K_h = K_m (1 - \phi)^3 / 2 \quad (4.19)$$

where:

K = conductivity of emulsion

K_m = conductivity of continuous phase, i.e. water

ϕ = in-situ volume fraction of oil

Equation (4.19) can be rearranged to give

$$\phi = 1 - \frac{(K_h)^{2/3}}{K_m} \quad (4.20)$$

Therefore, the in-situ volume fraction of oil can be determined from a knowledge of the conductivities of emulsions and the continuous phase, i.e. water.

8.3 Results and Discussion

Figure 8.1 shows the orifice meter data obtained for water and various concentrations of emulsions. The figure shows that the emulsion data follows the relationship obtained for pure water indicating that the water calibration can be used for oil-in-water emulsions. It is interesting to note that the data for the inverted

emulsion (the inversion of the O/W to the W/O emulsion occurred at the oil concentration of 77.5%) also followed the same relationship.

Figure 8.2 shows the venturi meter data obtained for water and the various emulsion concentrations. Here also the data for the emulsions follow the relationship obtained for single-phase water indicating that the water calibration may be used for both kinds of oil-in-water emulsions. The data for the inverted emulsion also follows the same relationship.

Figure 8.3 shows the magnetic flow meter data obtained for water and the various concentrations of emulsions. The line shown in the figure is drawn through the pure water data. The figure shows that the data for the O/W emulsion agree quite well with the water calibration. However, on inversion from O/W to W/O emulsion, where the oil in this case is totally non-conducting, the meter does not work. See the data plotted as stars which represents a 77.5% emulsion which has inverted.

Figure 8.4 shows the conductivity meter data obtained for water and the various emulsions. As the oil concentration increases, the conductance of the emulsion decreases. On inversion of the O/W emulsion to the W/O emulsion (which occurred at 77.5% oil concentration), the conductance of the emulsion jumped suddenly from a high value of 65 milli mho to 2 milli mho. For the inverted emulsion, the conductivity was also found to vary with the flow rate. The following table summarizes the conductivity meter results for the O/W emulsions:

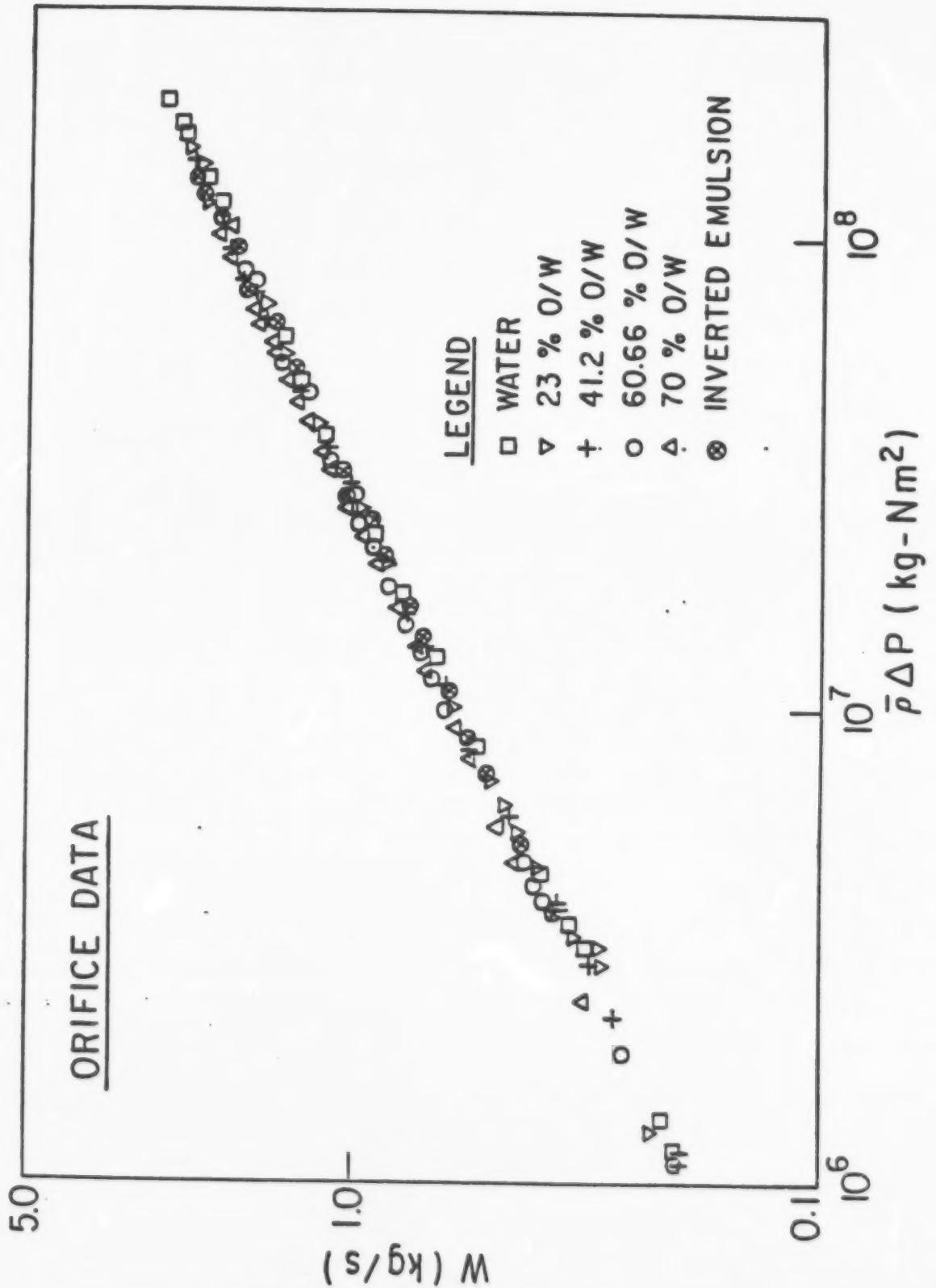


Figure 8.1: Performance of Orifice for O/W and Inverted Emulsions

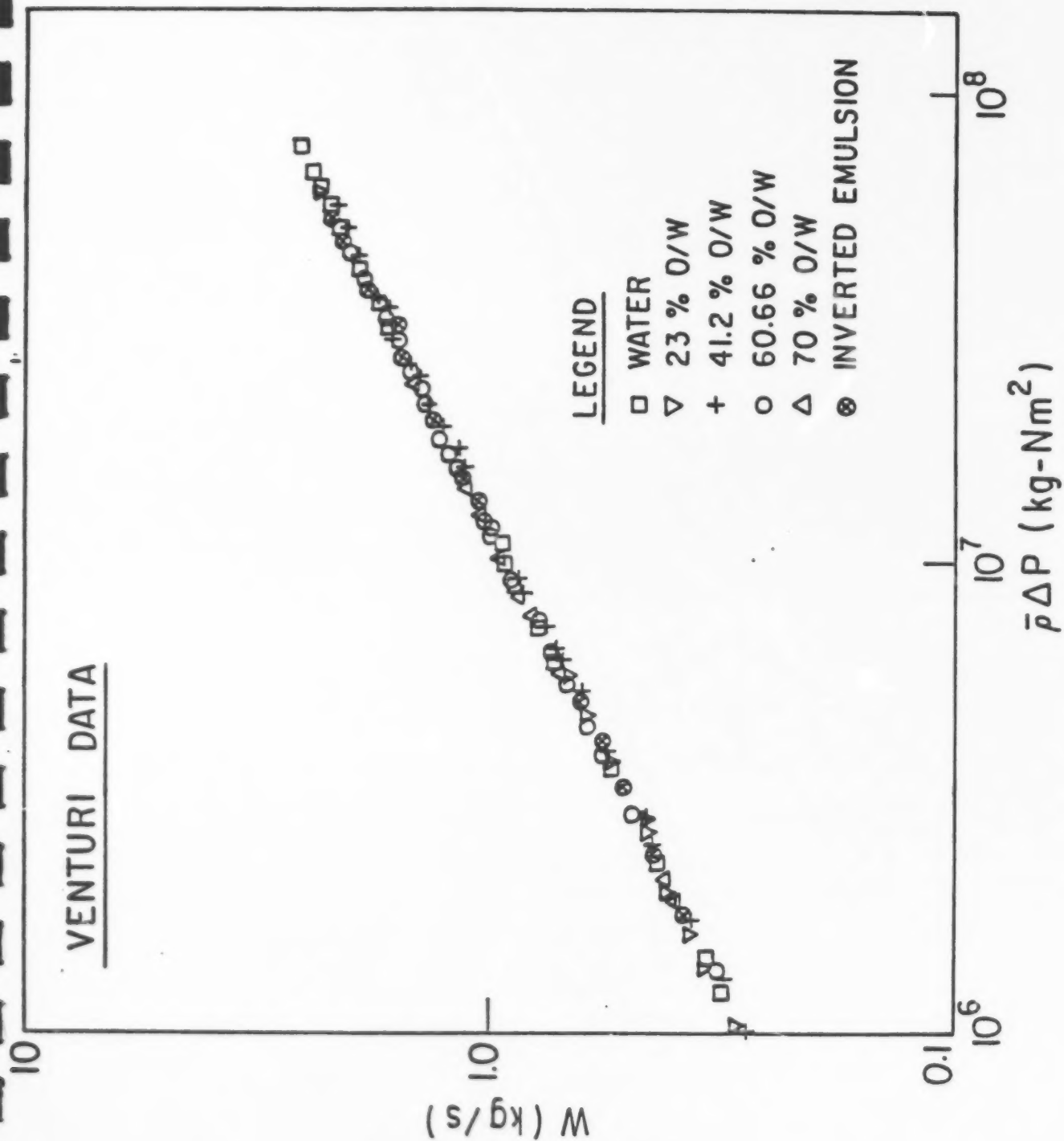


Figure 8.2: Venturi Data for O/W and Inverted Emulsions

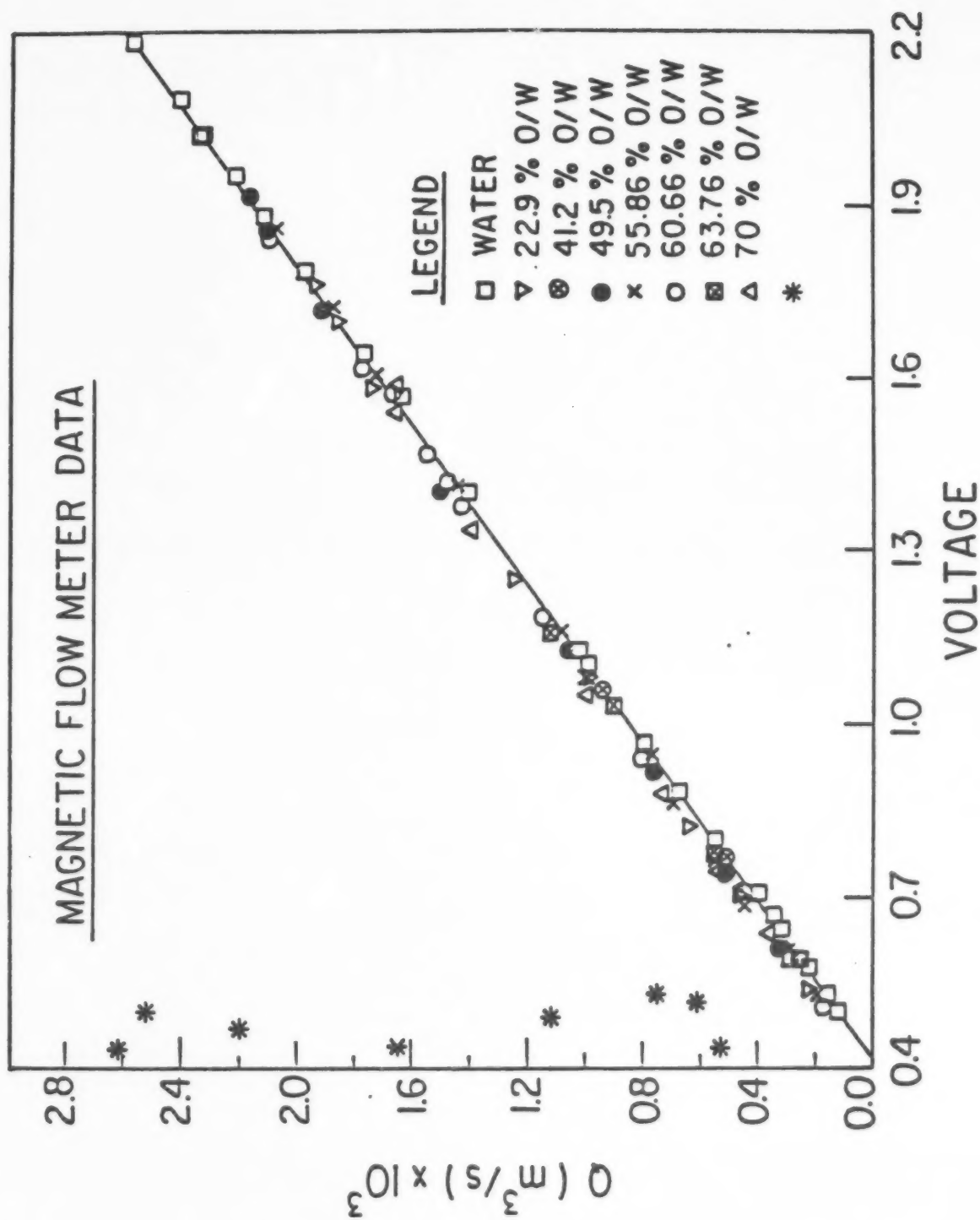


Figure 8.3: Magnetic Flow Meter Data for O/W Emulsions

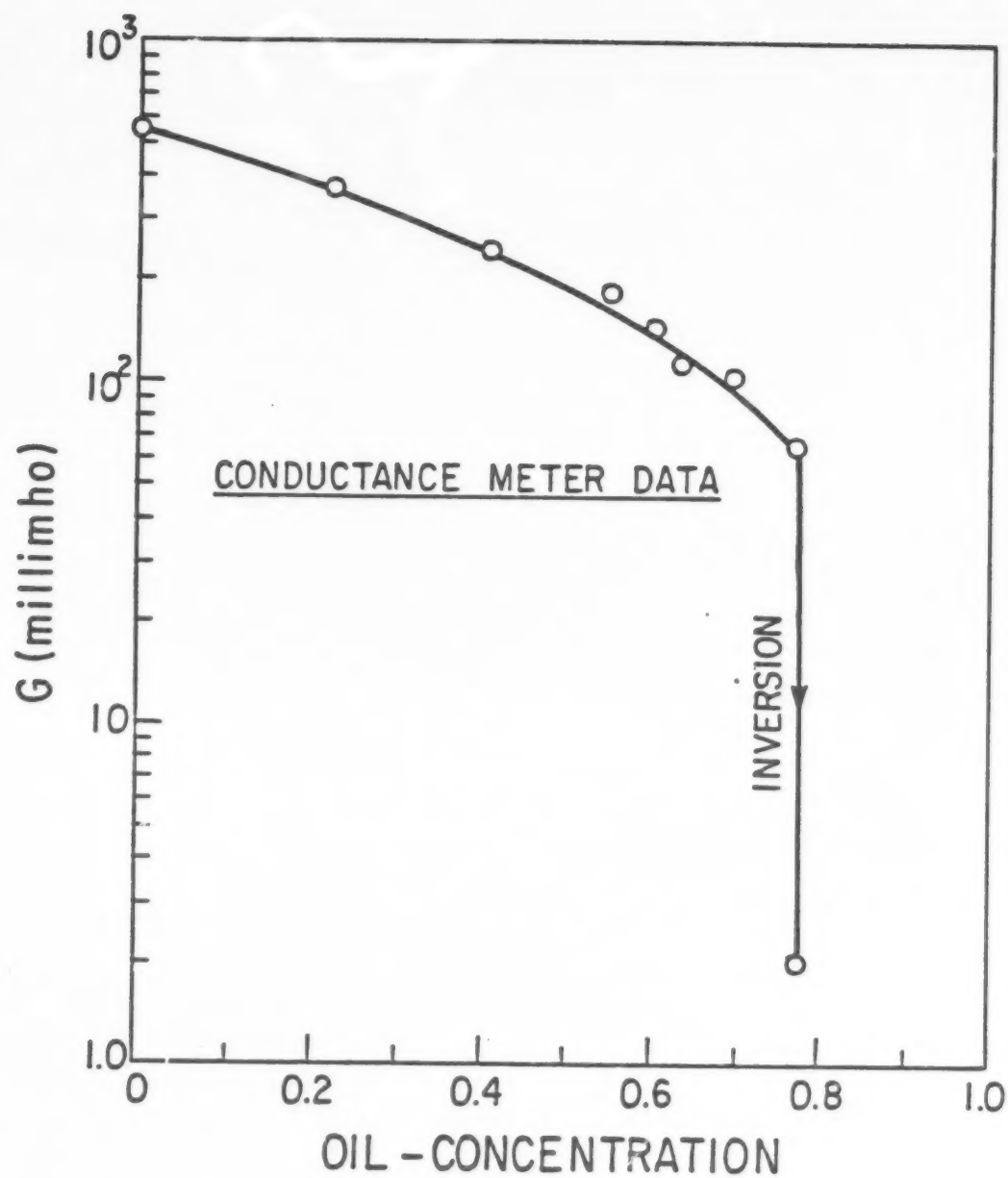


Figure 8.4: Conductivity Meter Data for O/W Emulsions

♦actual	Emulsion Conductance, milli mho	♦predicted from meter**
0	560	0
0.229	376.2	0.232
0.4119	246	0.422
0.5586	180	0.53
0.6066	142.5	0.598
0.64	111	0.65
0.70	101	0.68

** ♦ predicted from equation (4.20)

Obviously the conductivity meter probe used in these experiments is behaving according to the theoretical prediction except when the phases invert.

Chapter 9

ACHIEVEMENTS, CONCLUSIONS AND RECOMMENDATIONS

1. A detailed literature review of available technology on flow metering of oil-water emulsions produced in the heavy oil production industry shows that there are several claimed inventions of possible technology but few claims of successful application.
2. The Rheology of heavy oil dispersions is not well known or understood. No one has yet related the Rheological properties of emulsions to their pressure drop behaviours in pipelines.
3. An oil-water emulsion flow loop has been constructed in the laboratories of the University of Waterloo. This is a unique loop in Canada, and in future it will provide an opportunity for testing the flow characteristics of a wide variety of oil water emulsions. The loop contains pipelines of five different diameters and a return pipeline into which a broad spectrum of metering instruments can be inserted. The loop is instrumented with electrically operated pressure transducers, analog to digital converters, IBM micro-computer data acquisition equipment which has software capable of efficient data processing. It is strongly recommended that this equipment be used continuously in the coming years to generate flow data and measuring instrument data for both the Government and industry.

4. Preliminary studies of a mineral oil/water emulsion using the Weissenberg Rheogoniometer produced encouraging results. It was initially thought that the data would correlate well by using the so-called "maximum attainable concentration" as a parameter.

5. Subsequent work using two other kinds of viscometers, namely the Fann and the Brookfield types showed disparity in the data obtained from the three instruments. It was concluded that slip effects were affecting the correct results obtained from the Rheogoniometer and in subsequent work, the use of this instrument was abandoned.

6. A broad spectrum of Rheological data was correlated using a new parameter $\phi_{ur=100}$ which is the concentration at which an emulsion viscosity becomes one hundred times that of the continuous phase. It is recommended that this correlation be tested on a wide variety of oil/water emulsions of both synthetic and produced fluids and that this method be used to predict the performance of emulsion pipelines.

7. The Rheology of a real heavy oil was investigated using the Fann and Brookfield instruments. It was found that the pure heavy oil and its emulsions were greatly affected by aging and also by salt addition. It is recommended that these effects be studied in great detail in future projects.

8. The flow of unstable emulsions ranging in quality from 0 to 76% oil in water was investigated in five pipelines. It was found that the emulsions behaved like Newtonians (at least when flowing in

turbulent flow). An effective viscosity of the emulsion was determined from the pipeline data and it was found that it behaved according to the form of the Richardson correlation, i.e. $u_r = \exp(2.7\phi)$. A form of the Blasius equation correctly predicts the effective friction factor of the fluids flows in the pipeline provided the effective viscosity is used.

9. Using a mean density based on the oil/water concentration as measured by the conductivity meter, the orifice and venturi behaved in a very normal manner providing extremely good calibration curves for total mass flow. This performance extended to the situation where the emulsion inverted.

10. The magnetic flow meter also calibrated very well and of course had the added advantage of being independent of oil/water concentration. A drawback occurred when the phases inverted. Because the oil phase was non-conducting it was found that the magnetic flow meter gave no reading after inversion. It is recommended that the magnetic flow meter be further tested with bituminous oils because they do in fact have a conductivity and consequently, this may work in the magnetic flow meter. Certainly, the calibration curve is good when the continuous phase is continuous.

11. The conductance probe had a clear calibration when the continuous phase was conducting. When inversions occurred there was a sharp dip in the calibration curve making the use of the meter doubtful when the continuous phase is non-conducting. For bituminous oils this may not be the case and thus should be carefully studied.

12. A totally reliable and comprehensive metering system for all kinds of oil/water emulsions remains an elusive target for research engineers. The combination of the conductivity holdup meter with either the venturi, orifice or magnetic flow meter cannot yet be ruled out for bituminous oils.

Finally, in the Rheological part of the work, the properties of a heavy oil/water emulsion were studied in detail using the Fann coaxial cylinder viscometer. Based on the experimental data generated, the following conclusions were made:

- a. The heavy oil alone behaves like a Newtonian fluid but changes viscosity with aging.
- b. The O/W emulsion has a much higher viscosity than either of the single phases.
- c. On aging, the emulsions exhibit thixotropic behaviour.
- d. The viscosity of the emulsions changes considerably with the addition of sodium chloride.
- e. The effect of temperature is also to reduce the viscosity of the emulsion.

Further work needs to be done to establish generalized theories and practices for the classification and standardization of heavy oil emulsions. The effects of aging, for example, are very complex and difficult to predict.

NOMENCLATURE

- A = Constant in Eqn. (3.31)
 A_0 = Cross-section area of throat (or orifice)
 A_{in} = Some function of n' . (defined by eqn. 3.32 for power law fluids)
 C = Constant in Eqn. (3.31)
 C_D = Discharge coefficient, defined by Eqn. (4.20)
 C_n = Some function of n' (defined by Eqn. 3.33 for power law fluids)
 D_0 = throat (or orifice) diameter
 D = Diameter of tube
 e = Constant in Eqn. (3.310)
 f = Friction factor, defined by Eqn. (3.114)
 G_m = Electrical conductance of continuous phase
 K = Power law constant
 K' = Consistency index, defined by Eqn. (3.112)
 K_L = Electrical conductivity of dispersion at low frequency
 K_m = Electrical conductivity of continuous phase
 K_p = Electrical conductivity of dispersed phase
 K_h = Electrical conductivity of dispersion at high frequency
 K_d = Electrical conductivity of dispersion
 L = Length of tube
 n = Power law index
 n' = Flow behaviour index, defined by Eqn. (3.111)
 N_{Re} = Reynolds number, defined as $\rho DV/\mu$
 N'_{Re} = Generalized Reynolds number, defined by Eqn. (3.114)
 O/W = Oil-in-water emulsion

Δp = Pressure drop

Δp_0 = Pressure drop without drag reducing additive

$\Delta p_{\text{additive}}$ = pressure drop with additive

Q = volumetric flow rate

r = radial distance

R = tube radius

s = constant in Eqn. (3.310)

$u(r)$ = velocity (local) at distance

V = average velocity of fluid

W = mass flow rate

W/O = water-in-oil emulsion

GREEK LETTERS

β = ratio of throat (or orifice) diameter to pipe diameter, D_0/D

ϵ_d = dielectric constant of dispersion

ϵ_v = absolute dielectric constant of free space; 8.8541×10^{-14}
farad/cm

ϵ^* = complex dielectric constant of ($\epsilon^* = \epsilon_d - j\gamma_d$)

ϵ_m^* = complex dielectric constant of dispersion medium,
($\epsilon_m^* = \epsilon_m - j\gamma_m$)

ϵ_m = dielectric constant of dispersion medium

ϵ_p^* = complex dielectric constant of dispersed phase,
($\epsilon_p^* = \epsilon_p - j\gamma_p$)

ϵ_p = dielectric constant of dispersed phase

ϵ_l = dielectric constant of dispersion at low frequency

ϵ_h = dielectric constant of dispersion at high frequency

ϕ = dispersed phase volume fraction

γ_d = $K_d/\omega\epsilon_v$, imaginary part of the complex dielectric constant
of the dispersion

γ_m = $K_m/\omega\epsilon_v$, imaginary part of the complex dielectric constant
of the dispersion medium

γ_p = $K_p/\omega\epsilon_v$, imaginary part of the complex dielectric constant
of the dispersed phase

γ = shear rate (= $-du/dr$ in tubes)

ν = viscosity

ν_∞ = viscosity at infinite shear rate

ν_t = turbulent viscosity

ω = measuring frequency

ρ = density

ρ_m = density of continuous phase

ρ = mean density of dispersion ($\rho = \phi \rho_p + (1-\phi) \rho_m$)

ρ_p = density of dispersed phase

τ = shear stress

τ_w = shear stress at the tube wall

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APPENDIX 1 - PUBLICATIONS

The following papers have been prepared for possible publication in journals and conferences.

Paper 1: "Metering of Two Phase Liquid-Liquid Flow".

R. Pal and E. Rhodes. Proceedings of the Third Multiphase Flow and Heat Transfer Symposium Workshop. Elsevier Press 835-849 (1984) - preliminary work done on flow metering before commencement of contract.

Paper 2: "Measuring Oil and Water Production of Wells".
R. Pal and E. Rhodes, Review paper.

Paper 3a: "Slip Effects in Flow of Concentrated Emulsions".
R. Pal, E. Rhodes and N.A. Mansour. Intended for CSChE Journal.

Paper 3b: "Slip Effects in Flow of Concentrated Emulsion".
R. Pal and E. Rhodes. Extended abstract of paper presented at 34th CSChE Conference, Quebec, October 1984.

Paper 4: "Flow Behaviour of Oil-in-Water Emulsions".
R. Pal, S.N. Bhattacharya and E. Rhodes. A condensed version of this paper was presented at the International Congress on Rheology, Mexico (see paper 5). It is intended to submit this paper to CSChE Journal.

Paper 5: "Rheological Study of Concentrated Emulsions".
R. Pal, S.N. Bhattacharya and E. Rhodes. To be presented at IX International Congress on Rheology, Acapulco, Mexico, October 8-13 (1984).

Paper 6: "A Novel Viscosity Correlation for Non-Newtonian Concentrated Emulsions".
R. Pal and E. Rhodes. Intended for the J. Colloid and Interface Science.

Paper 6b: "Viscosity Correlation for Non-Newtonian Concentrated Emulsions".
R. Pal and E. Rhodes. Uncut version of paper intended for the J. of Colloid and Interface Science.

Paper 7: "On the Rheology of Heavy Oil and its Emulsions".
R. Pal and E. Rhodes. Intended for the Canadian J. Petroleum Technology.

PAPER 1

METERING OF TWO PHASE LIQUID-LIQUID FLOW

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ABSTRACT

The monitoring of oil and water in a horizontal oil-in-water (O/W) emulsion flow was performed using an in-line flow meter. The metering system consisted of a conductivity meter downstream of a Herschel-type venturi meter. The conductivity meter enabled oil hold up to be determined and the venturi meter enabled the total mass flow rate to be determined.

The metering experiments were performed with white mineral oil ($\rho_p = 0.844 \text{ gm/cc}$, $\mu = 15 \text{ cp}$ at 27°C) and water at room temperature ($26.5 \pm 0.5^\circ\text{C}$). The conductivity of water was varied between 100 milli mho/m and 230 milli mho/m by adding sodium chloride. The mass flow rate was varied from 12 kg/min to 42 kg/min and the oil hold up was varied from 0 to 0.7.

A relationship was found between the conductivity of emulsion and the volume fraction of the oil phase. A homogeneous flow model for the venturi meter was found to fit the experimental data well in the range of flow rates studied. The results obtained from the experiments indicated that this metering system should be acknowledged as a feasible and efficient O/W emulsion flow meter.

INTRODUCTION

Emulsions are mixtures of two immiscible liquids, one of them being dispersed in the other in the form of globules. There may exist either an oil-in-water (oil is the disperse phase) or a water-in-oil (water is the disperse phase) emulsion. These emulsion types are conveniently abbreviated O/W and W/O, respectively. Familiar examples of these systems can be found in everyday life and particularly in the oil industry.

As crude oil rises from the fissures of the earth, its passage through narrow openings, accompanied by water and agitation by pumping, gives rise to conditions favourable to the formation of W/O emulsions.

The transportation of highly viscous crude oils in the form of O/W emulsions has received special attention in the recent years. The viscosity and hence the pumping energy requirements of crude oils is greatly reduced by forming an O/W emulsion. Two commercial emulsion pipeline systems for crude transport are known. One in Indonesia [1] carries 40,000 barrels/day of a 70 percent (volume) emulsion of a high pour point waxy crude in a 20 inch diameter line which is 238 kilometers long. The other, in California [2], carries a 50 percent emulsion of highly viscous 13.5 API gravity crude thirteen miles in an 8-inch line.

Under some conditions, one type of emulsion will invert or change into the other type. McAuliffe et al. [3] were able to invert the highly viscous, naturally produced W/O emulsions into the much more fluid O/W ones by using a "Down-the-Hole" method, in which the chemicals (non-ionic surfactants) were pumped down into the well.

A large number of patents [4-9] are available in the literature related to the flow measurement of W/O emulsions. However, there is little or no published data on continuous monitoring of oil and water in oil-in-water emulsion flow. The purpose of this study was to develop a metering technique to monitor oil and water continuously in a horizontal O/W emulsion flow.

THEORY

Conductivity

The electrical conductivity of the mixture of oil and water is a reliable index (since the conductivity of oil is significantly less than that of water) of the proportions of oil and water in the mixture. It depends upon several factors [10]:

1. Type of emulsion (O/W or W/O)
2. Conductivities of continuous and dispersed phases
3. Volume concentration of dispersed phase
4. Measuring frequency
5. Temperature

Emulsions in which water is the continuous medium (O/W type) show a high conductivity; emulsions in which oil is the continuous medium (W/O type) may be expected to show little, or no, conductance. In general, the conductivity of O/W emulsions increases with the temperature and decreases with the increase in volume concentration of the dispersed phase (oil).

Considerable theoretical and empirical work has been done in correlating the conductivity of emulsions with some of the variables mentioned above. For a dispersion of spherical particles in a liquid

at low concentrations, Wagner [11] derived the following relationship between the complex dielectric constant of the dispersion ϵ^* and the volume fraction:

$$\frac{\epsilon^* - \epsilon_m^*}{\epsilon^* + 2\epsilon_m^*} = \frac{\epsilon_p^* - \epsilon_m^*}{\epsilon_p^* + 2\epsilon_m^*} \phi \quad (1)$$

Substituting the relations for ϵ^* , ϵ_p^* , ϵ_m^* (refer to definitions) and separating the real and imaginary parts, Eq. 1 reduces to:

(i) At low frequencies ($\omega \rightarrow 0$)

$$\frac{K_g}{K_m} = \frac{2K_m + K_p - 2\phi(K_m - K_p)}{2K_m + K_p + \phi(K_m - K_p)} \quad (2)$$

(ii) At high frequencies ($\omega \rightarrow \infty$)

$$\frac{K_h}{K_m} = \frac{K_m(2\epsilon_m + \epsilon_p)^2 + [(9K_p - 2K_m)\epsilon_m^2 - 8K_m\epsilon_m\epsilon_p + K_m\epsilon_p^2]\phi - 2K_m(\epsilon_m - \epsilon_p)^2\phi^2}{[2\epsilon_m + \epsilon_p + \phi(\epsilon_m - \epsilon_p)]^2} \quad (3)$$

In the special case for O/W emulsions:

$$K_m \gg K_p \text{ and } \epsilon_m \gg \epsilon_p$$

Eq. 2 reduces to

$$\frac{K_g}{K_m} = \frac{2(1 - \phi)}{2 + \phi} \quad (4)$$

and Eqn. 3 reduces to

$$\frac{K_h}{K_m} = \frac{2(1 - \phi)}{2 + \phi} \quad (5)$$

From Eqs. 4 and 5:

$$K_2 = K_h \quad (6)$$

Wagner's theory does not hold for concentrated dispersions, since this theory was derived for a dilute dispersed system. Hanai [12-14] developed a theory for a concentrated dispersed system on the basis of Wagner's theory. Hanai made the following assumptions:

1. A concentrated dispersed system can be obtained by successively adding infinitesimally small quantities of dispersed phase into the dispersion medium.
2. Wagner's relation (Eq. 1) holds for this process with infinitesimally small change in concentration.

Based on the above assumptions, Hanai derived the following relationship:

$$\frac{\epsilon^* - \epsilon_p^*}{\epsilon_m^* - \epsilon_p^*} \left(\frac{\epsilon_m}{\epsilon^*} \right)^{1/3} = 1 - \phi \quad (7)$$

Substituting the relations for ϵ^* , ϵ_p^* , ϵ_m^* (refer to definitions) and separating the real and imaginary parts Eq. 7 reduces to:

(i) At low frequencies ($\omega \rightarrow 0$)

$$\frac{K_l - K_p}{K_m - K_p} \left(\frac{K_m}{K_l} \right)^{1/3} = 1 - \phi \quad (8)$$

(ii) At high frequencies ($\omega \rightarrow \infty$)

$$K_h \left[\frac{3}{\epsilon_h - \epsilon_p} - \frac{1}{\epsilon_h} \right] = 3 \left[\frac{K_m - K_p}{\epsilon_m - \epsilon_p} + \frac{K_p}{\epsilon_h - \epsilon_p} \right] - \frac{K_m}{\epsilon_m} \quad (9)$$

In the special case for O/W emulsions:

$$K_m \gg K_p, \epsilon_m \gg \epsilon_p, K_l \gg K_p, K_h \gg K_p \text{ and } \epsilon_h \gg \epsilon_p$$

Eq. 8 reduces to

$$\frac{K_l}{K_m} = (1 - \phi)^{3/2} \quad (10)$$

and Eq. 9 reduces to

$$\frac{K_h}{K_m} = (1 - \phi)^{3/2} \quad (11)$$

From Eqs. 10 and 11,

$$K_L = K_h \quad (12)$$

2.2 Venturi

The mass flow rate of a single phase fluid through the venturi meter is a function of the pressure drop across the venturi, the fluid density and the dimensions of the system according to the venturi equation [15].

$$W = \frac{C S_0}{\sqrt{1 - R^4}} \sqrt{2\rho \Delta p} \quad (13)$$

For a well designed venturi meter, the coefficient C is about 0.98 for pipe diameters of 2 to 8 inch and about 0.99 for larger sizes if the pipe Reynolds number is above 10^5 .

Homogeneous flow model [16]. If it is assumed that the dispersion behaves as a single phase fluid, the following equation (i.e. homogeneous flow model) can be used to determine the mass flow rate through the venturi meter.

$$W = \frac{C S_0}{\sqrt{1 - R^4}} \sqrt{2 \rho \Delta p} \quad (14)$$

For fully dispersed (homogeneous) slurry flow [17] in pipes it is known that the friction factor very nearly approaches that for the dispersion medium. Thus one would expect C for such flows to approach the corresponding values for the pure fluid.

3. EXPERIMENTAL APPARATUS

The experimental apparatus used in this study is shown schematically in Figure 1. The dispersion was prepared in a 60 gallon baffled polyethylene tank. The agitation of the emulsion was accomplished by a 1750 r.p.m. Philadelphia portable mixer. Circulation of the emulsion to the test section was provided by a centrifugal pump equipped with a by-pass to regulate the flow rate.

The metering system consisted of a venturi meter upstream of an in-line conductivity meter. The venturi meter was entirely constructed out of transparent acrylic. The inside diameter of the venturi was 2.54 cm and 1.5875 cm at the throat. Two pressure taps were located on the venturi section; one just upstream of the contraction and one at the throat. The pressure taps consisted of four 0.1587 cm holes drilled into the acrylic tubing and equally

spaced around the circumference. All these holes were contained in a 1.016 cm wide piezometer ring containing a 0.9525 NPT connecting hole. This ring was sealed to the tube using two "O" rings. ; A detailed diagram of the venturi is given in Figure 2.

Differential pressure readings were relayed to a pressure transducer. The transducer signals were routed to a digital readout. The pressure range for the transducer was 0 - 6.89 kPa.

The conductivity meter unit consisted of a conductivity cell wired to a standard conductivity meter (Figure 3). The conductivity cell was connected 35 cm downstream of the venturi meter. The main body of the conductivity cell was a cylindrical pipe made out of transparent acrylic. Eight metal pieces, equally spaced around the circumference, were fixed at the inside of the cylindrical pipe so as to form a star shaped electrode system.

4. EXPERIMENTAL WORK

Before the commencement of the two phase experimental work, the calibration of an in-line conductivity cell and the venturi meter was done with water alone. The conductivity of water was varied between 35 milli mho/m and 1000 milli mho/m by adding sodium chloride. The conductivity of water in the tank was measured by a conductivity cell of known cell constant of 10/m.

A calibration curve (Figure 4) was established by plotting the conductance of water, G_m , (measured by an in-line conductivity cell) against the conductivity of water, K_m , measured by the conductivity cell of known cell constant.

To prepare the dispersions, a known volume of oil was added to a previously known volume of water in the mixing tank. The conductivity of water was checked before mixing with the oil. The two liquids were then mixed thoroughly with a propeller mixer for about 30 minutes. The dispersion had a milky-white appearance, characteristic of many liquid-liquid emulsions. The agitation was maintained throughout the experimental run.

At start up, the pressure tap lines were purged with water to ensure that no air pockets were trapped. The flow rates, adjusted by using a by-pass valve on the pump, were measured by diverting the flow to a calibrated tank, using a quick change-over valve mechanism. The pressure drop reading from the transducer and the reading from an in-line conductivity cell were recorded for each flow rate.

The experiments were performed at room temperature ($26.5 \pm 0.5^\circ\text{C}$). The oil hold up range of 0 to 0.7 was investigated. The total mass flow rate was varied from 12 kg/min to 42 kg/min.

5. FLOW METERING RESULTS

5.1 Venturi Meter

The calibration data for the venturi meter are shown in Figure 5, where the mass flow rate W is plotted versus the term $\bar{\rho} \Delta p$. In the range of flow rates studied, the coefficient C is nearly constant and the relationship between $\log W$ and $\log (\bar{\rho} \Delta p)$ is linear. The data for various emulsions show good agreement with the line which represents single phase water. Thus the assumptions that the dispersion behaves as a single phase fluid and the coefficient C approaches the values corresponding to the dispersion medium appear justified. Hence the homogeneous flow model (Eq. 14) may be used to determine the flow rate

of emulsions.

5.2 Conductivity Meter

In Figure 4, a calibration curve for an in-line conductivity cell is presented. It is obtained by plotting the conductance of water (G_m) measured by an in-line meter against the conductivity of water (K_m) measured by a conductivity cell of known cell constant.

A plot of an in-line conductivity meter reading (G) versus the oil hold up (ϕ) with water conductivity as a parameter is shown in Figure 6. The conductance of the emulsions decreases with the increase in the oil hold up and increases with the increase in the conductivity of continuous medium (water). The effect of flow rate on an in-line conductivity meter reading is found to be negligible. Only at very low flow rates, G decreases probably because of the separation of oil and water phases.

In Figure 7 is shown the relationship between the relative conductivity (K/K_m) and the oil hold up (ϕ). The relative conductivity decreases with the increase in the oil hold up. When $\log K/K_m$ is plotted against $\log (1 - \phi)$ a linear relationship is obtained (Figure 8). There is a good agreement between the experimental data and the theoretical prediction (Eq. 10).

5.3 Two Phase Flow Metering

Oil hold up is determined from the conductance measurements. It is calculated from

$$K/K_m = (1 - \phi)^{3/2} \quad (15)$$

where K is the conductivity of the mixture measured by an in-line conductivity meter and K_m is the conductivity of the continuous medium (water). Figure 9 shows a plot of $\phi_{\text{meter}}/\phi_{\text{measured}}$ versus ϕ_{measured} . ϕ_{meter} is determined from Eq. 15 and ϕ_{measured} is known from the experiments (it is the ratio of the volume of oil to the total volume of the mixture in the emulsion tank). The deviation of ϕ_{meter} from ϕ_{measured} is generally within 4% except for low values of ϕ_{measured} .

Total mass flow rate of emulsion is calculated from a knowledge of oil hold up and pressure drop across the venturi meter using a homogeneous flow model (Eq. 14). The accuracy is within 6% of the true value.

The oil mass flow rate, W_o is determined from

$$W_o = xW \quad (16)$$

where x is the quality of the mixture defined as

$$x = [\rho_o \phi] / [\rho_o \phi + \rho_w (1 - \phi)] \quad (17)$$

In Figure 10, a plot of $W_{o,\text{meter}}/W_{o,\text{measured}}$ versus ϕ_{measured} is presented. $W_{o,\text{meter}}$ is the oil mass flow rate calculated from Eq.

16 using Eqs. 14, 15 and 17. $W_{O,measured}$ is known from the experiments (found by collecting a certain volume of the fluid and recording the time of collection). The accuracy is within 7% except for low values of $\phi_{measured}$.

6. CONCLUSIONS

The conclusions of this study are the following:

1. A homogeneous flow model for the venturi meter can be used to determine the total mass flow rate of O/W emulsions through the meter. The accuracy is within 6% of the true value.
2. The conductivity meter can be used to find the proportions of oil and water in the two phase mixture. A linear relationship exists between $\log K/K_m$ and $\log (1 - \phi)$. The deviation of ϕ from the true value is generally within 4%.
3. The coupling of a conductivity meter with a venturi meter is a feasible in-line, oil-in-water emulsion flow metering system. Oil mass flow rate, as measured by this system, is within 7% of the true value.

ACKNOWLEDGEMENTS

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DEFINITIONS

$\epsilon^* = \epsilon - jY$, complex dielectric constant of the dispersion

$\epsilon_m^* = \epsilon_m - jY_m$, complex dielectric constant of the dispersion
medium

$\epsilon_p^* = \epsilon_p - jY_p$, complex dielectric constant of the dispersed
phase

$Y = K/\omega\epsilon_v$, imaginary part of the complex dielectric constant of the
dispersion

$Y_m = K_m/\omega\epsilon_v$, imaginary part of the complex dielectric constant
of the dispersion medium

$Y_p = K_p/\omega\epsilon_v$, imaginary part of the complex dielectric constant
of the dispersed phase

ϕ = volume fraction of oil in emulsion or oil hold up

$\bar{\rho} = \phi \rho_p + (1 - \phi)\rho_m$, mean density of the dispersion

$x = [\rho_o \phi]/[\rho_o \phi + (1 - \phi)\rho_w]$, quality or weight fraction of oil
in the two phase mixture

NOTATION

C = venturi coefficient, dimensionless

G = conductance of the dispersion, milli mho

G_m = conductance of the dispersion medium, milli mho

K = conductivity of dispersion, milli mho/m

K_h = limiting conductivity of dispersion at high frequency, milli
mho/m

K_l = limiting conductivity of dispersion at low frequency, milli
mho/m

K_m = conductivity of dispersion medium, milli mho/m

K_p = conductivity of the dispersed phase, milli mho/m

Δp = pressure drop across venturi, N/m^2

S_0 = cross sectional area of the throat of the venturi meter, m^2

w = measuring frequency, cycles/sec

W = total mass flow rate, kg/sec

W_o = oil mass flow rate, kg/sec

x = weight fraction of oil in the two phase mixture, dimensionless

Y = imaginary part of the complex dielectric constant of the dispersion, dimensionless

Y_m = imaginary part of the complex dielectric constant of the dispersion medium, dimensionless

Y_p = imaginary part of the complex dielectric constant of the dispersed phase, dimensionless

Greek Letters

B = ratio of throat diameter to pipe diameter, dimensionless

ϵ = dielectric constant of the dispersion, dimensionless

ϵ^* = complex dielectric constant of the dispersion, dimensionless

ϵ_h = limiting dielectric constant of dispersion at high frequency, dimensionless

ϵ_l = limiting dielectric constant of dispersion at low frequency, dimensionless

ϵ_m = dielectric constant of the dispersion medium, dimensionless

ϵ_m^* = complex dielectric constant of the dispersion medium, dimensionless

ϵ_p = dielectric constant of the dispersed phase, dimensionless

ϵ_p^* = complex dielectric constant of the dispersed phase,
dimensionless

ϵ_v = absolute dielectric constant of free space, 8.8541×10^{-14}
farad/cm

μ = viscosity, cp

ϕ = volume fraction of oil in emulsion or oil hold up, dimensionless

$\bar{\rho}$ = mean density of the dispersion, kg/m^3

ρ_m = density of the dispersion medium, kg/m^3

ρ_p = density of dispersed phase, kg/m^3

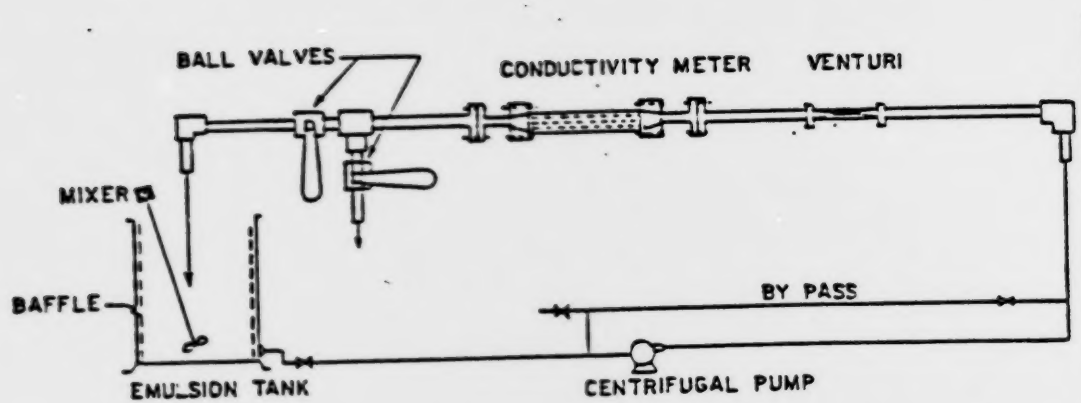


Fig. 1. Schematic Drawing of the Experimental System

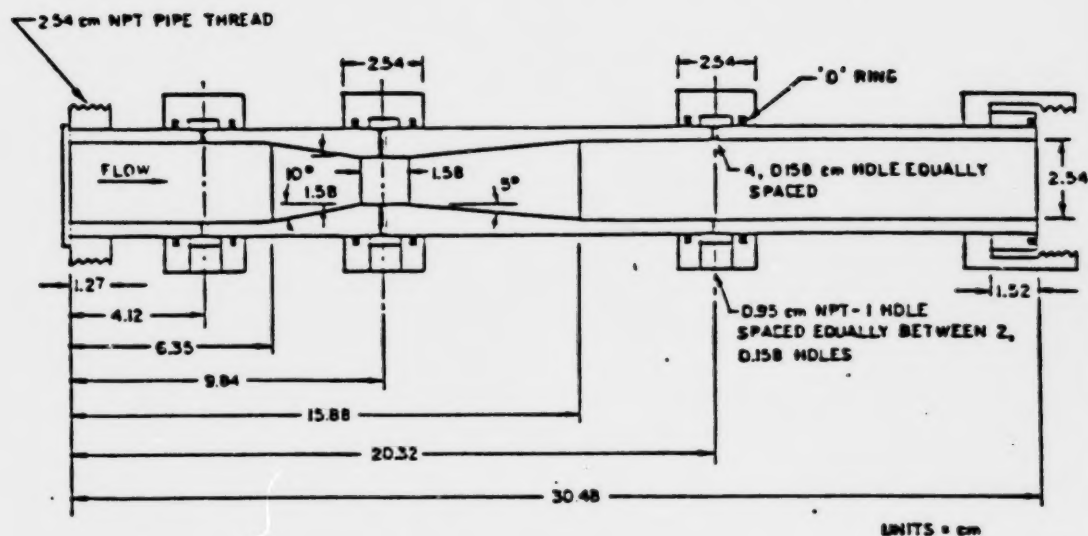


Fig. 2. Venturi meter

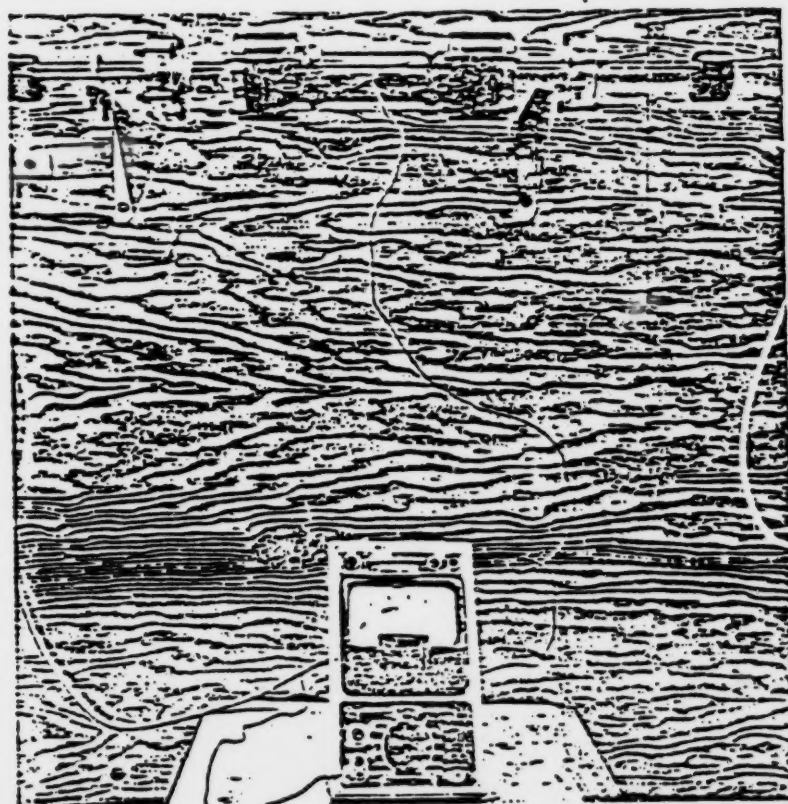


Fig. 3. Conductivity meter

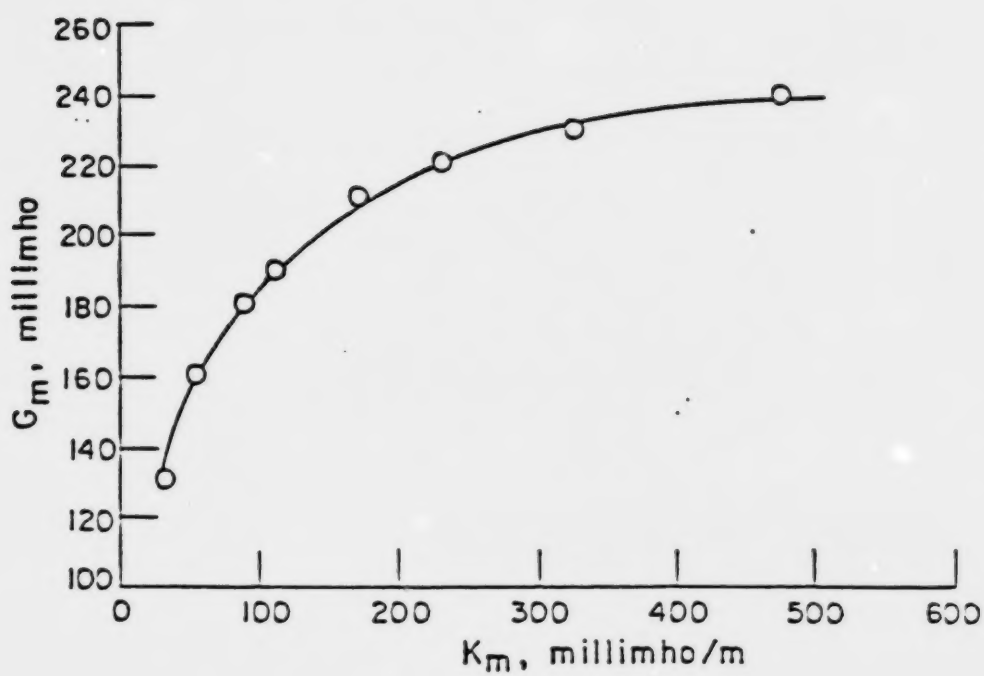


Fig. 4. Conductivity Cell Calibration

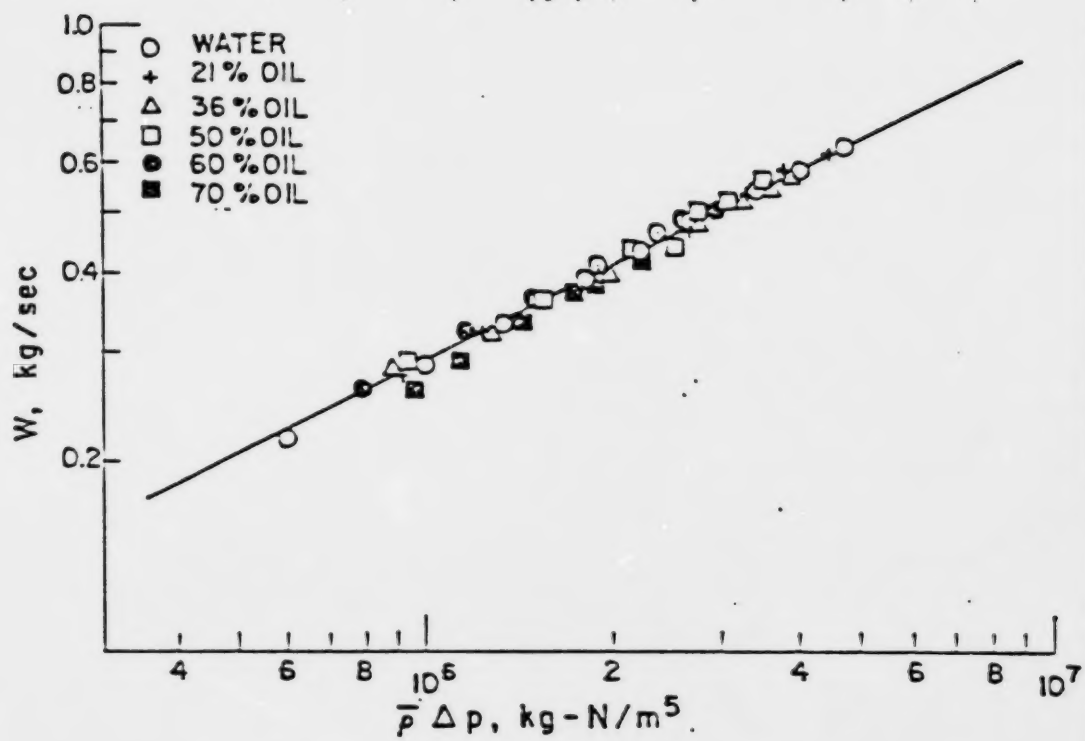


Fig. 5. Venturi Calibration

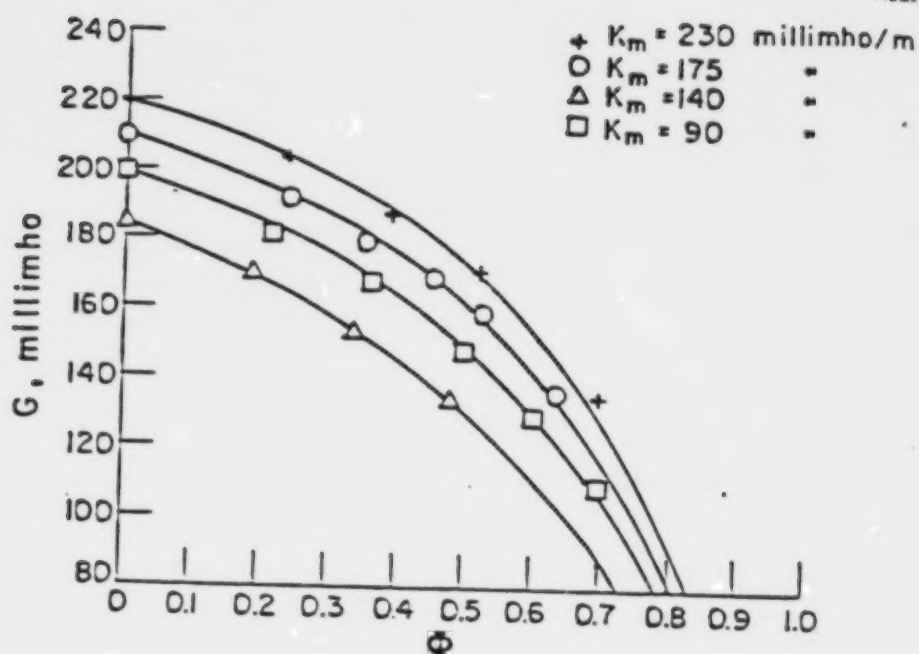


Fig. 6. Concentration dependence of conductance for the emulsions

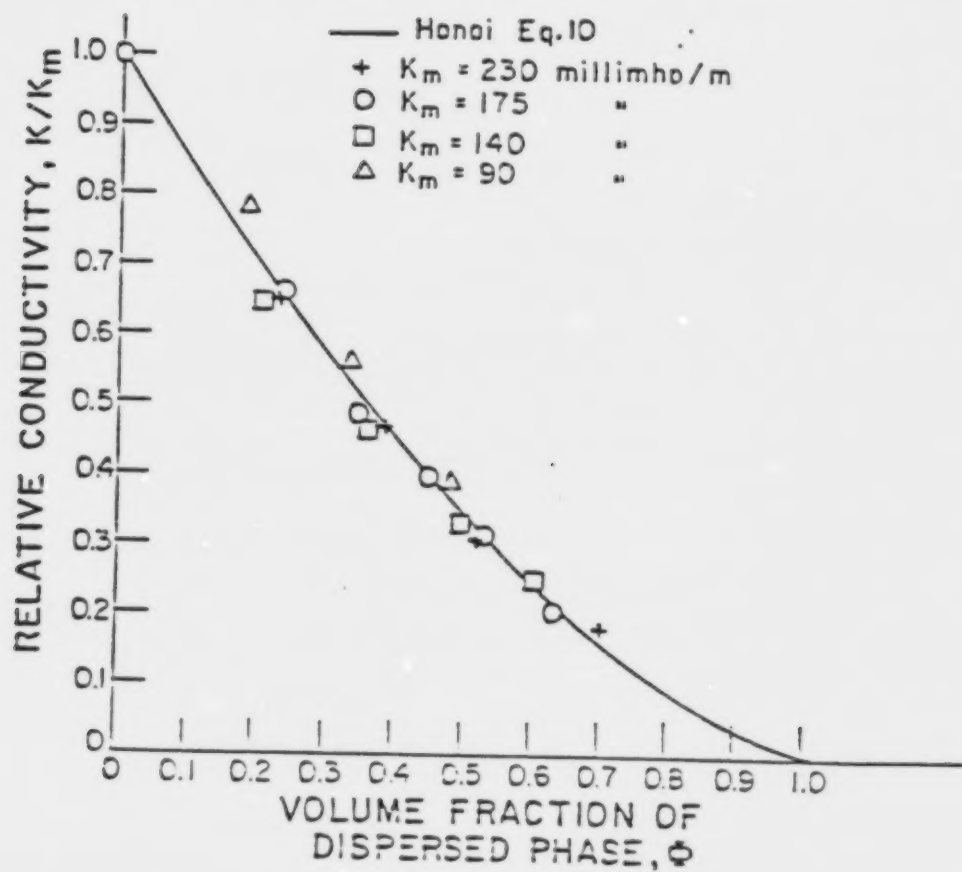


Fig. 7. Concentration dependence of electrical conductivity for the emulsions

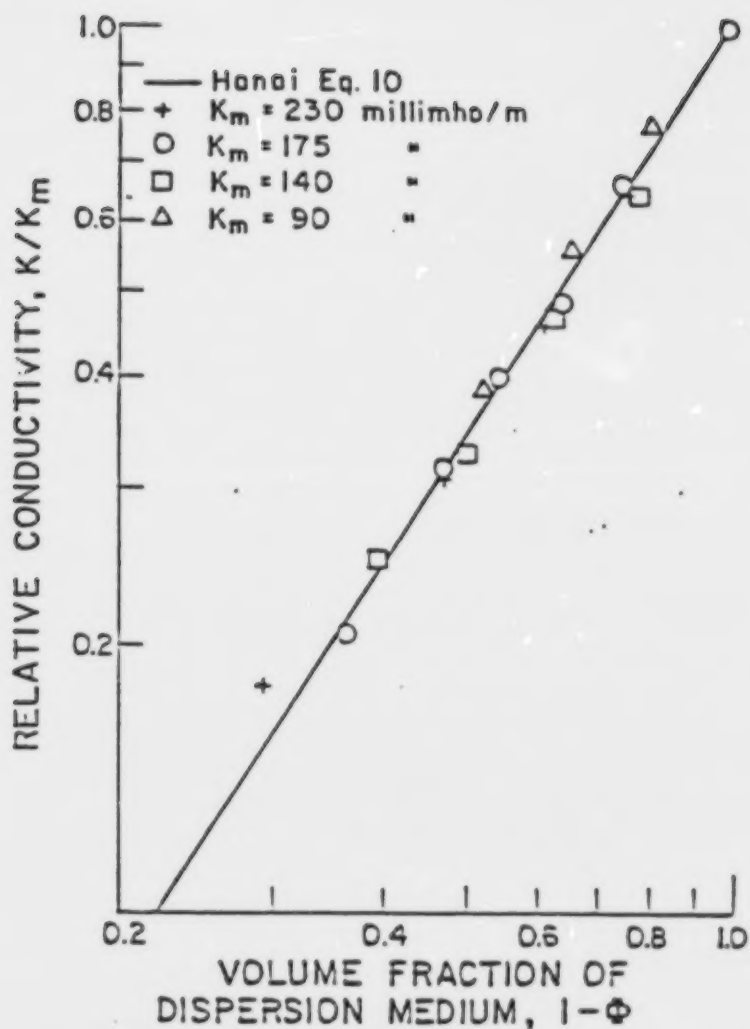


Fig. 8. Dependence of relative conductivity on the volume fraction of dispersion medium

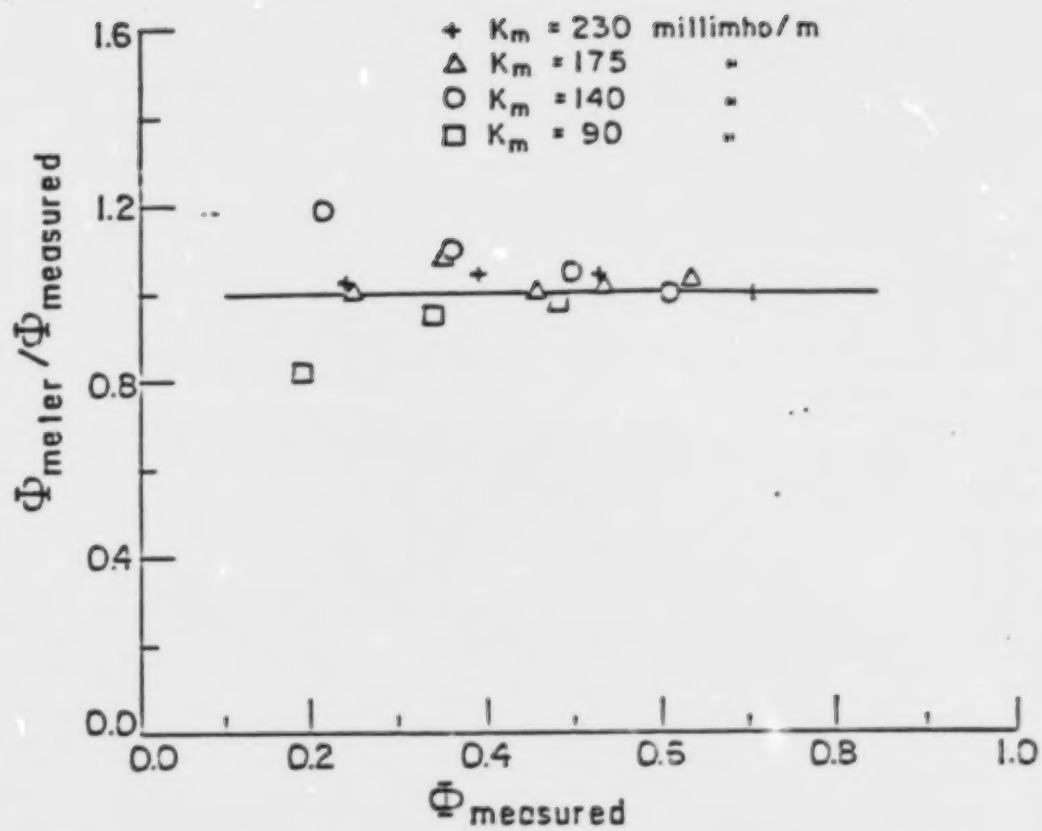


Fig. 9. Oil hold up given by the meter vs. the true oil hold up

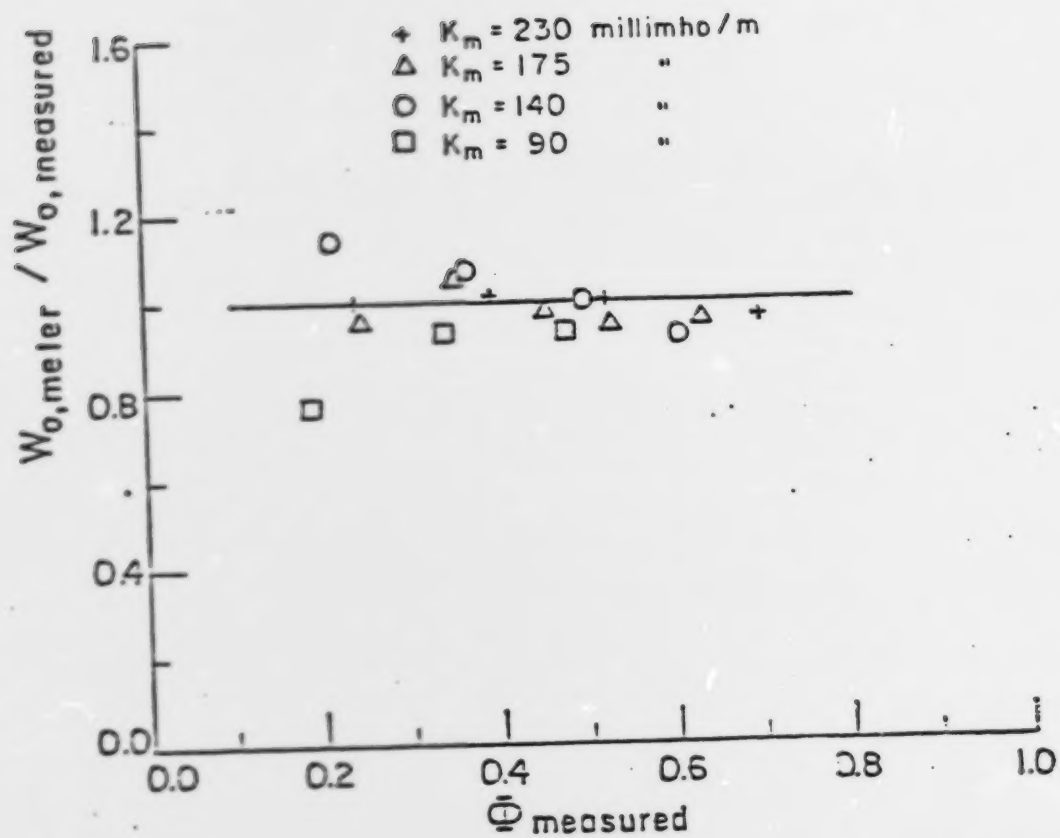


Fig. 10. Oil mass flow rate given by the meter vs. the oil hold up

PAPER 2

MEASURING OIL AND WATER PRODUCTION OF WELLS

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ABSTRACT

Methods for continuous monitoring of oil and water in the flow of water-in-oil (W/O) emulsions are described. A new metering system is described that can be used to meter oil-in-water (O/W) emulsions. Advantages and disadvantages of various metering techniques are discussed.

On-line measurement of a two phase W/O mixture requires two measurements: volume fraction (sometimes called the "Water-cut") of the dispersed phase and the total flow rate. These two measurements are combined to get separate flow rates of oil and water in a pipeline. "Water-cut" or volume fraction of water in water-in-oil emulsions is measured by either a Capacitance probe, Vortex meter, Densitometer probe, or a Differential Pressure Unit (DPU). Total flow rate is measured by a turbine flow meter. The "water-cut" meter is coupled with the total flow meter to separate the flow rates of oil and water.

Oil hold up or volume fraction of oil in W/O emulsions is measured by an in-line conductivity meter. Total flow rate is measured by a venturi meter. The conductivity meter is coupled with a venturi meter to separate the oil and water flow rates.

INTRODUCTION

The production from oil wells often includes water which is combined with the oil in the form of a water-in-oil (W/O) emulsion [1]. The high viscosity of these W/O emulsions frequently limits the rate at which crude oil can be produced from a well. It causes low pump volumetric efficiency, slow rod dropping rate and high flow line pressure drop. To overcome these limitations [2-6], surface active chemicals are added to the well to convert high viscosity oil or water-in-oil (W/O) emulsions to low viscosity oil-in-water (O/W) emulsions.

In view of the above mentioned typical application, it is desirable to have an on-line, instantaneous measurement of oil and water flow rates in the flow of W/O and O/W emulsions. In this paper, we will review the techniques that are used in the oil industry to meter W/O emulsions and describe a new technique that can be used to meter O/W emulsions. Advantages and disadvantages of the various techniques will be discussed.

W/O EMULSIONS

Water-cut: This is defined as the volume fraction of water in a W/O emulsion. The instruments which have been used to measure the water-cut are the Capacitance probe, Vortex meter, Densitometer probe and Differential Pressure Unit (DPU).

Capacitance probe [7-14]: The capacitance probe continuously measures the dielectric constant of the mixture of oil and water. As the water content of the mixture changes, the dielectric constant as measured by the probe also changes. The dielectric constant of the mixture is a reliable index of the proportions of oil and water since the dielectric constant of oil is significantly less than that of water. The dielectric constant of water might

typically be 80 and the oil may have a typical value of 2. The limiting dielectric constant at high frequencies ($\omega \rightarrow \infty$) for W/O emulsions is related to water-cut by the following equation [15-18]:

$$\frac{\epsilon_p - \epsilon_h}{\epsilon_p - \epsilon_m} \left(\frac{\epsilon_m}{\epsilon_h} \right)^{1/3} = 1 - \phi \quad (1)$$

Since $\epsilon_p \gg \epsilon_m$ and $\epsilon_p \gg \epsilon_h$, equation (1) reduces to

$$\frac{\epsilon_h}{\epsilon_m} = (1 - \phi)^{-3} \quad (2)$$

A capacitance probe consists of two electrodes, one electrode may take the form of a tubular metallic element having an outer diameter almost equal to the inner diameter of a pipeline. The other electrode may take the form of a rod which is positioned axially in the centre of a pipeline by means of non-conductive spiders. However, any other suitable electrode system may be used. The signal from the capacitance probe is converted into frequency or a voltage analog directly proportional to the dielectric constant of the mixture.

Vortex meter [19-22]: The vortex meter utilizes a vortex generating object which is placed at a suitable location in the flow stream. This object produces oscillating pressure pulses (due to vortex shedding) at a frequency proportional to the volumetric flow rate. The amplitude of these pulses is proportional to the mass flow rate. The Strouhal number N_s , is indicative of the frequency at which vortex shedding occurs and is a function of the Reynolds number. For a wide range of Reynolds number, the Strouhal number is constant. The Strouhal number is defined as

$$N_s = f \frac{D}{u_m} \quad (3)$$

where f = frequency of vortex formation
 u_{∞} = free stream velocity
 D = effective diameter of the object

In the range of Reynolds number for which N_S is constant

$$u = K_1 f \quad (4)$$

or

$$Q = K_2 f \quad (5)$$

The amplitude of the pressure pulse is a function of the kinetic energy contained in the vortex

$$A = K_3 \rho u^2 / 2 = K_4 f^2 \rho \quad (6)$$

or

$$\frac{A}{f} = K_4 f \rho = K_5 \rho Q \quad (7)$$

Therefore, the frequency of the output signal gives the volumetric flow rate and the amplitude divided by the frequency of the signal gives the total mass flow rate of the mixture. The mean density signal is generated by dividing the mass flow signal by the volumetric flow signal. The calibration of the meter does not depend on fluid properties and is determined only by the dimensions of the flow element and the pipeline. The concentration of water or "water-cut" is determined from:

$$\phi = \frac{\rho - \rho_0}{\rho_w - \rho_0} \quad (8)$$

Therefore if the densities of the oil and water phases are known, then a water cut can also be calculated.

Two types of vortex meters are available commercially, one is the swirl meter type and the other is bluff body, vortex shedding type. In the swirl meter type, fluid is forced to assume a swirl motion. At the inlet section of the flow tube there is a fixed set of swirl blades which impart a swirling motion to the fluid passing through. There is a venturi section downstream of the swirl blades. The venturi section first constricts and then expands the flow to transform the swirling motion into processional movement in the expanding region of the venturi section to create a vortex. In the bluff body type meter, a vortex generating object is placed in the flow stream transversely with respect to the flow axis to generate fluidic oscillations whose frequency is proportional to the flow rate.

Densitometer probe [23-25]: The densitometer probe continuously measures the mean density of the mixture of oil and water in the pipeline. A thin rectangular stainless steel vane is made to vibrate in the fluid at its natural resonant frequency. This natural frequency is dependent upon the total mass of the oscillating system (which includes the vane and a fixed volume of the surrounding fluid) and therefore, an increase in fluid density results in a lowering of the frequency of oscillation. For calculation of density, we may consider an equivalent system represented by a hollow body of mass M (mass of the vane) which is suspended on a spring with an elasticity constant k , its volume V (volume of the fluid taking part in the oscillation) filled with a sample of density ρ . The natural frequency of this system will be

$$f = \frac{1}{2\pi} \sqrt{\frac{k}{M + \rho V}} \quad (9)$$

The period of oscillation, T is defined as:

$$T = \frac{1}{f} = 2\pi \sqrt{\frac{M + \rho V}{k}} \quad (10)$$

Taking the square of this expression and inserting

$$A' = \frac{4\pi^2 V}{k} \quad B' = \frac{4\pi^2 M}{k} \quad (11)$$

we obtain

$$T^2 = A' \rho + B' \quad (12)$$

or

$$\rho = \frac{1}{A'} [T^2 - B'] \quad (13)$$

The constants A' and B' are regarded as apparatus constants which may be determined from two calibration measurements of samples of known density. If ρ_r is some known fixed density for a period T_r , then

$$\rho_r = \frac{1}{A'} [T_r^2 - B'] \quad (14)$$

subtracting equation (14) from equation (13)

$$\rho - \rho_r = \frac{1}{A'} [T^2 - T_r^2] \quad (15)$$

Therefore by measuring the period of oscillation, the mean density of the two phase oil-water mixture is determined. Water-cut or the concentration of water in W/O emulsion is then determined from equation (8). The probe (Figure 1) consists of a shank, a housing at its upper end, a tubular assembly at its lower end and an electrical connector assembly at the upper end of the housing. The tubular assembly of the probe is mounted coaxially in the pipeline by using annular fittings around the shank. The vane, which is inserted in the tubular

assembly in a plane parallel with the direction of flow, is vibrated by a magnetostrictive driver. A piezoelectric crystal is the pick up, the output of which is converted by means of an electronic circuit into a current and/or voltage analog proportional to the density of the mixture.

Differential pressure unit [26-28]: This system, like the densitometer probe, measures the mean density of the fluid flowing in a pipeline. A pressure difference between two points at different elevations in the pipe is measured and this pressure differential is proportional to the mean density of the two-phase oil and water mixture. A Differential Pressure Unit (DPU) consists of two inlet tubes (Figure 2) A and B maintained at different elevations in a pipeline. Bellows are used at the inlet of these tubes to keep oil-water mixture on the right side of the bellows separate from the fluid on the left side. Pressure in the pipeline is transmitted through the fluid in these tubes to another pair of bellows. By a common linkage the differential pressure effect on the two bellows produces a corresponding rotation of the shaft. This rotative motion of the output shaft of a differential fluid pressure transducer is converted to a corresponding electric signal analog (which is proportional to the mean density of oil and water mixture flowing in a pipeline). The mean density of the mixture and the "water-cut" are related to each other by equation (8).

Total Flow Rate [9-14, 23-27]

Most of the reported net oil flow computers utilize a turbine flow meter to determine the total volumetric flow rate, Q , of W/O emulsions. A turbine flow meter consists of a straight flow tube containing a free running rotor, mounted coaxially within a pipe, which rotates with an angular velocity proportional to the actual velocity of flow in the pipe. As the velocity of flow is directly

proportional to the volumetric flow rate, the speed of rotation is proportional to the volumetric flow rate. The flow meter has a magnetic or other pick-up unit suitable for detecting the rotation of the rotor in response to the flow rate of fluid through the conduit. The output signal of the flow meter constitutes a series of electrical pulses. The pulse repetition frequency (PRF) of the pulses is directly proportional to the volume flow rate within pipeline.

Separate flow rates of oil and water in a pipeline are determined by combining the total flow signal and the "water-cut" signal:

$$Q_w = \phi Q \quad (16)$$

$$Q_o = (1 - \phi)Q \quad (17)$$

O/W EMULSIONS

Oil-Hold-up: is defined as the volume fraction of oil in a two phase O/W emulsion can be measured by an in-line conductivity meter.

Conductivity meter [29]: Conductivity meter continuously measures the conductivity of oil-in-water emulsion. As the oil content of the mixture changes, the conductivity also changes. The conductivity of the mixture is a reliable index of the proportions of oil and water since the conductivity of oil is significantly less than that of water. The limiting conductivity [15-18] of oil-in-water emulsions at low frequencies ($\omega \rightarrow 0$) is related to the oil hold up as:

$$\frac{K_e - K_p}{K_m - K_p} \left(\frac{K_m}{K_e} \right)^{1/3} = 1 - \phi \quad (18)$$

The limiting conductivity at high frequencies ($\omega \rightarrow \infty$) is given as:

$$K_h \left[\frac{3}{\epsilon_h - \epsilon_p} - \frac{1}{\epsilon_h} \right] = 3 \left[\frac{K_m - K_p}{\epsilon_m - \epsilon_p} + \frac{K_p}{\epsilon_h - \epsilon_p} \right] - \frac{K_m}{\epsilon_m} \quad (19)$$

where

$$\frac{\epsilon_h - \epsilon_p}{\epsilon_m - \epsilon_p} \left(\frac{\epsilon_m}{\epsilon_h} \right)^{1/3} = 1 - \phi \quad (20)$$

Since $\epsilon_m \gg \epsilon_p$, $\epsilon_h \gg \epsilon_p$, $K_m \gg K_p$, $K_l \gg K_p$ and $K_h \gg K_p$ equation (18) reduces to;

$$\frac{K_l}{K_m} = (1 - \phi)^{3/2} \quad (21)$$

equation (20) reduces to;

$$\frac{\epsilon_h}{\epsilon_m} = (1 - \phi)^{3/2} \quad (22)$$

equation (19) reduces to;

$$\frac{K_h}{\epsilon_h} = \frac{K_m}{\epsilon_m} \quad (23)$$

From equations (22) and (23)

$$\frac{K_h}{K_m} = (1 - \phi)^{3/2} \quad (24)$$

From equations (21) and (24)

$$K_h = K_l \quad (25)$$

Therefore, the conductivity of O/W emulsions is independent of the measuring frequency. By measuring the conductivity of an emulsion and the continuous phase, oil-hold up is determined from equation (21). In Figure 3 are shown the results of conductivity measurements done by Pal & Rhodes [29]. The relative conductivity, defined as the ratio of the conductivity of emulsion (K) to the conductivity of continuous phase (K_m), is plotted as a function of oil-hold up on log-log scale. As oil-hold up increases, the relative conductivity decreases. There is an excellent agreement between the experimental data and the theoretical prediction (Eq. 21). The deviation of ϕ calculated from Eq. 21 is generally within 4 percent of the true value. The conductivity cell used by Pal & Rhodes (shown in Figures 4 and 5) consists of a star shaped electrode system. However any other suitable electrode system could be used. The flow area inside the cell is kept the same as the flow area upstream and downstream of the cell to reduce the head losses.

Total Flow Rate [29]

The total mass flow rate of an O/W emulsion can be determined by a venturi meter. Assuming that the dispersion behaves as a single phase fluid, the homogeneous flow model is used to determine the mass flow rate through the venturi meter.

$$W = \frac{C S_0}{\sqrt{1 - \beta^4}} \sqrt{2\rho \Delta p} \quad (26)$$

where C is the venturi coefficient if the dispersion medium alone is flowing at the same mass flow rate as the emulsion. The venturi meter calibration data obtained by Pal & Rhodes (reference 29) for various concentrated emulsions ($21\% < \phi < 70\%$) are shown in Figure 6. The coefficient c is nearly constant in the range of flow rates studied and the relationship between $\log w$ and $\log \rho \Delta p$

is linear. Emulsions show good agreement with the line which represents single phase water. The oil mass flow rate, W_o is determined from:

$$W_o = x W$$

where x is the quality of the mixture defined as:

$$x = [\rho_o \phi] / [\rho_o \phi + \rho_w (1 - \phi)]$$

The deviation of oil mass flow rate W_o , as calculated from Eq. 27, is generally within 7% of the true value.

DISCUSSION

A capacitance probe coupled with a turbine flow meter is the most common technique used in the oil industry to meter W/O emulsions. Current Hydrill and CE Natco net oil computers are based on this principle. The important advantage of using the capacitance principle to measure the concentration of water (and therefore of oil) is that the dielectric constant of water phase is only a weak function of salinity, and since there is such a big difference between the dielectric constants of oil and water, the salinity has very little effect on the instrument readings. The other advantage is that the capacitance probe is relatively simple in construction. However, the capacitance probe utilized in such systems is operative only for W/O emulsions. When there is inversion of the W/O emulsion to an O/W one (which can occur if water content of the mixture is high) the capacitance probe will tend to short out due to electrode polarization and will provide an erroneous indication of the amount of oil in the fluid.

Techniques based on the mean density of two phase oil-water mixture (Vortex meter, Densitometer probe and DPU) may not be so useful since, quite often, the difference in the densities of oil and water is not large. In the case of a 10 percent (water concentration) W/O emulsion with $\rho_o = 0.9$ gm/cc and $\rho_w =$

1.0 gm/cc, one percent error in the mean density signal will give 90 percent error in "water-cut". The other disadvantage with these systems is that they are relatively more complicated and expensive in construction.

Turbine flow meters have found widespread use in the oil industry. They have the advantage of having digital and practically linear output over a fair range of flow rates. Also they produce only moderate head loss. The calibration characteristic of turbine meters is sensitive to changes in the viscosity of liquid being metered. The effect of viscosity is great in small turbine meters, becoming less serious as the size of meter increases. Since W/O emulsions are non-Newtonian (viscosity changes with the flow rate) it is difficult to obtain high accuracy. Calibration of these meters is also affected by gradual wear or fouling of surfaces and therefore periodic recalibration is necessary if accuracy is to be maintained. Turbine meters are rather more expensive than many types of flow meter, particularly in the larger diameters.

A conductivity meter coupled with a differential pressure meter is a feasible and relatively inexpensive in-line O/W emulsion metering system. Its great advantage is the application to O/W emulsion metering. Because the difference in conductivities between oil and water is very large, the requirements for the conductivity meter are by no means critical. However, the full extent of the application of this proposed technique needs to be investigated by using stable emulsions and a wide range of flow rates. Further investigations should involve continuous monitoring of the conductivity of the continuous medium (water).

ACKNOWLEDGEMENTS

Appreciation is expressed to CAMET for a research contract which enabled this study to proceed.

NOMENCLATURE

A	amplitude of the pressure pulse in Eq. (6)
A', B'	constants defined by Eqs. (11)
C	venturi coefficient
D	diameter, m ²
f	frequency, cycles/sec
k	constant in Eq. (9)
K ₁	constant in Eq. (4)
K ₂	constant in Eq. (5)
K ₃ , K ₄	constants in Eq. (6)
K ₅	constant in Eq. (7)
K _h	limiting conductivity of emulsion at high frequency, milli mho/m
K _l	limiting conductivity of emulsion at low frequency, milli mho/m
K _m	conductivity of continuous phase, milli mho/m
K _p	conductivity of dispersed phase, milli mho/m
M	mass of the vibrating vane, kg
N _s	Strouhal Number
O/W	oil-in-water emulsion
Δp	pressure drop, N/m ²
Q	total volumetric flow rate of emulsion, m ³ /sec
Q _o	volumetric flow rate of oil, m ³ /sec
Q _w	volumetric flow rate of water, m ³ /sec
S _o	cross sectional area of the throat of the venturi, m ²
T	period of oscillation, sec
T _r	period of oscillation with fluid of known density, sec
u	average fluid velocity, m/sec
u _∞	free stream velocity, m/sec

V	volume of the fluid taking part in oscillation, m^3
W/O	water-in-oil emulsion
W	total mass flow rate of emulsion, kg/sec
W_o	oil mass flow rate, kg/sec
x	quality of emulsion defined by Eq. (28)

Greek Symbols

β	ratio of throat diameter of venturi to pipe diameter
ϵ_h	limiting dielectric constant of emulsion at high frequency
ϵ_m	dielectric constant of continuous phase
ϵ_p	dielectric constant of dispersed phase
ϕ	volume fraction of the dispersed phase
ω	angular frequency, radians/sec
ρ	mean density of emulsion, kg/m^3
ρ_o	density of oil, kg/m^3
ρ_r	density of reference fluid, kg/m^3
ρ_w	density of water, kg/m^3

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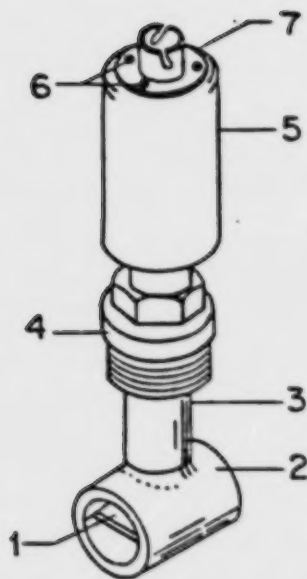


Figure 1. Densitometer probe (1) Vane, (2) Tubular assembly, (3) Shank, (4) Annular fitting, (5) Housing, (6) Bolts, (7) Electrical connector assembly.

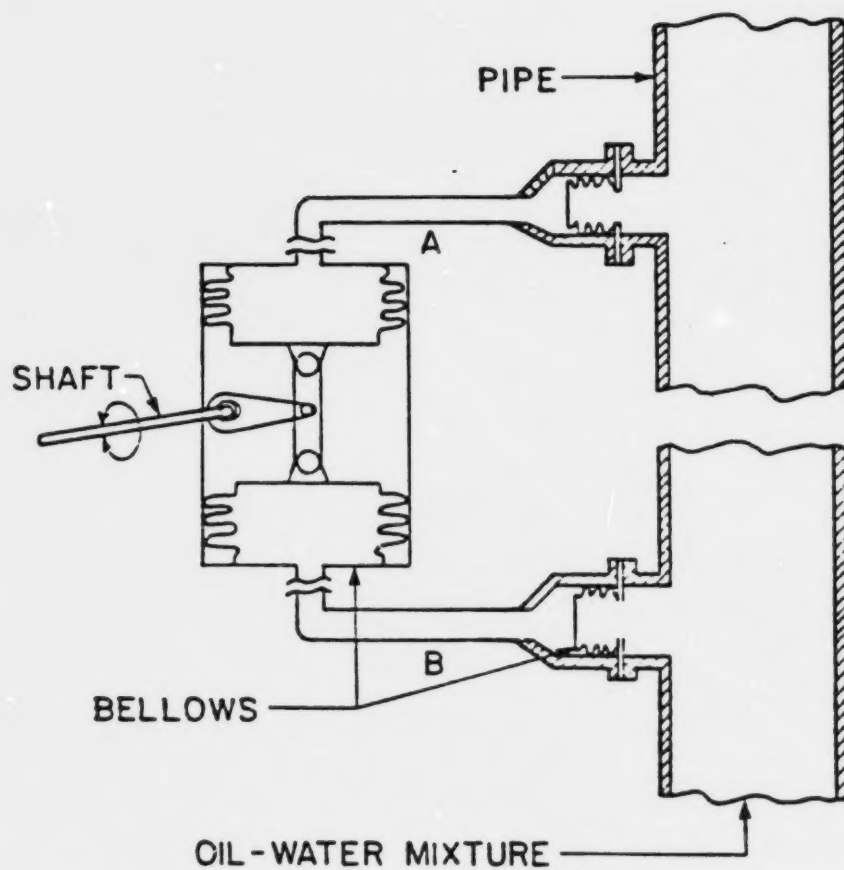


Figure 2. Differential pressure unit (DPU)

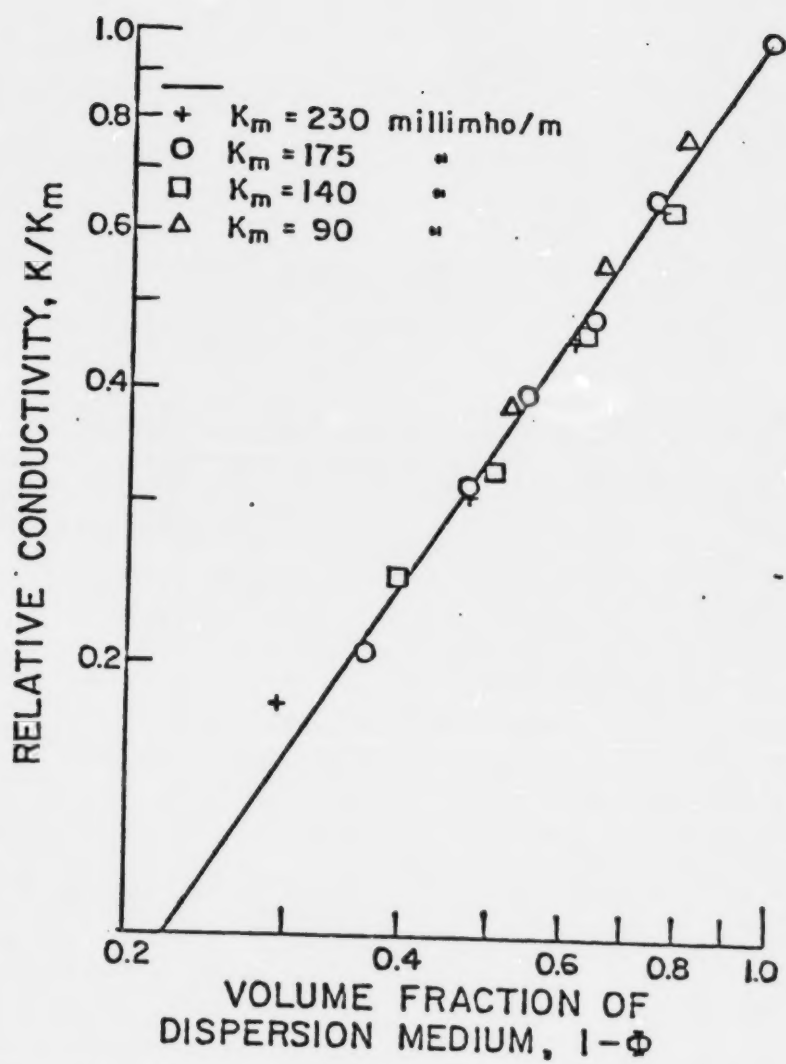


Fig. 3 Dependence of relative conductivity on the volume fraction of dispersion medium

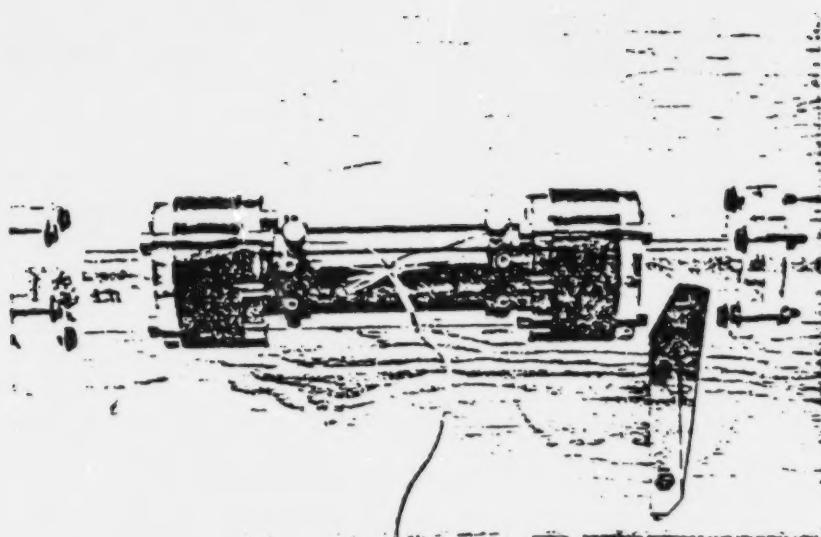


Figure 4. Conductivity cell

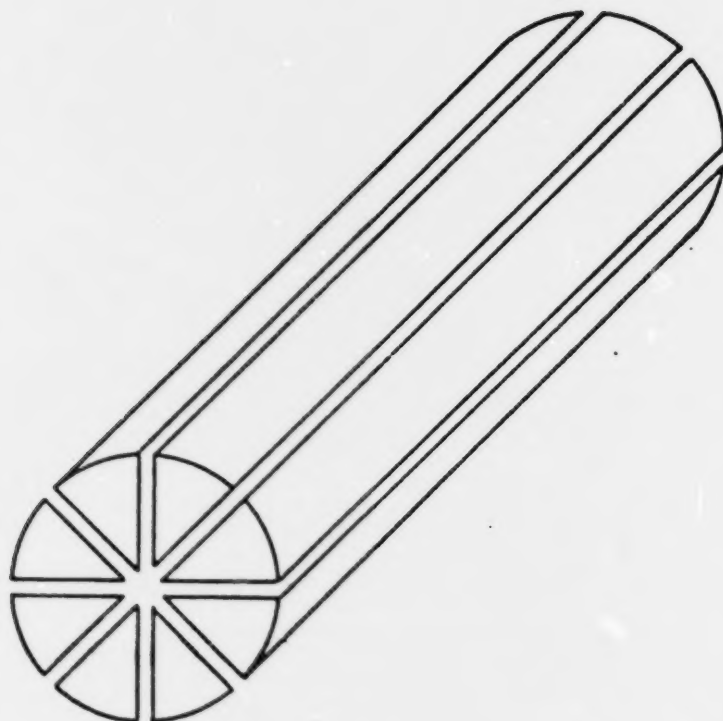


Figure 5. Internal view of conductivity cell: eight metal pieces are fixed around the circumference of the pipe to form star shaped electrode system

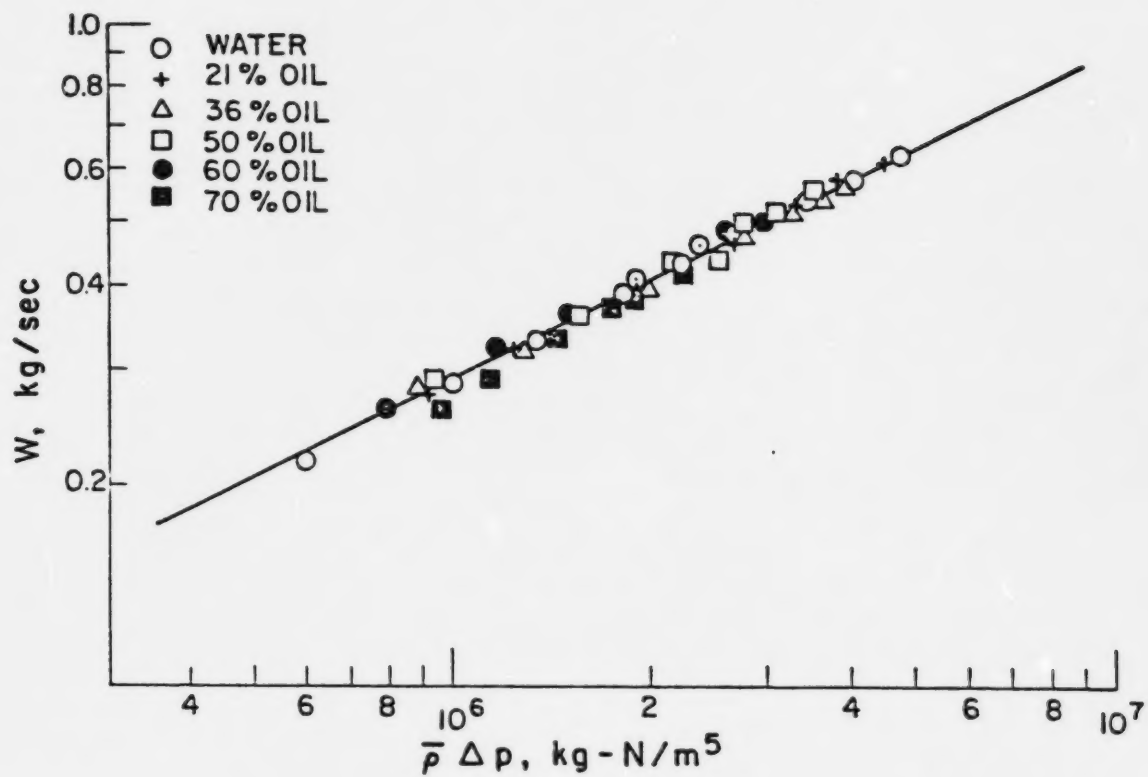


Fig. 6 Venturi Calibration

PAPER 3a

"SLIP EFFECTS" IN CONCENTRATED EMULSIONS

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ABSTRACT

In this study, rheological data obtained for various concentrated oil-in-water and water-in-oil emulsions from viscometers of different design and geometry have been analyzed and compared. The three types of viscometers used were the; FAN viscometer (coaxial cylinder type); Weissenberg Rheogoniometer (cone-and-plate type) and Brookfield viscometer (rotating bob in an infinite fluid).

In the case of oil-in-water emulsions, the flow curves obtained with the cone-and-plate viscometer are in close agreement with those given by coaxial cylinder viscometer only when the oil concentrations are below 70%. For concentrations greater than 70%, the flow curves obtained from the two instruments are appreciably different.

In the case of water-in-oil emulsions the flow curves obtained from different instruments are in agreement only when water phase concentrations are below 60%. For water concentrations greater than 60%, the flow curves obtained are significantly different.

The variation in the flow curves for the same concentration emulsions has been attributed to the presence of "slip-effects" in the cone-and-plate viscometer.

INTRODUCTION

A considerable amount of the world's crude oil is produced in the form of stable emulsions (1). Water-in-oil types of emulsions are mainly encountered in primary production. On the other hand, oil-in-water emulsions (also known as reverse emulsions), are more frequently encountered in secondary and tertiary oil recovery projects. Many situations exist (2-7) where oil-in-water emulsions are deliberately made in order to reduce the high viscosity (and hence pumping cost) of crude oils. The use of emulsions to enhance oil recovery is receiving special attention in current years (8).

To evaluate the rheological properties of emulsions, instruments of many designs are available commercially. They fall into four main categories, viz: rotating bob in an infinite fluid, coaxial cylinder, cone-and-plate and capillary.

To ensure that the rheological properties determined are the properties of the emulsion and not artifacts produced by the method of investigation, it is very important and often necessary to obtain comparative data from viscometers of different designs. If different viscometers do not give the same rheological measurements, then it may be unwise to test the emulsions in simple viscometers and use the results to predict the behaviour of the same emulsion in pipelines, mixing equipment, etc. The main objective of this study was to analyze and compare emulsion rheological data obtained from viscometers of different designs and determine if "slip effects" were present. The emulsions used in the study were made up to represent emulsions which might be encountered in the heavy oil recovery industry.

EXPERIMENTAL

Emulsion Preparation:

Oil-in-water emulsions were prepared by the agent-in-water method at room temperature ($\sim 24^{\circ}\text{C}$). The oil phase was 73.3% by volume (white mineral oil) dissolved in 26.7% by volume carbon-tetrachloride which was added to the mineral oil in order to make the density of oil phase equal to that of water phase. In this way the creaming effect in the emulsions was eliminated. One percent by volume (based on the water phase) of non-ionic emulsifier Triton x-100, was added to deionized water and this mixture was slowly stirred until the emulsifier was completely dissolved. To this mixture of water and emulsifier, the required amount of the oil phase was slowly added and the mixture was sheared in a high shear machine (Gifford-Wood Model 1-LV Homogeneizer-Mixer) for about 15 minutes. To obtain more highly concentrated emulsions, more oil phase was added to the existing lower concentration emulsion and the mixture sheared again for 15 minutes. In the 76% emulsion, some air entrapment was noted and therefore the next higher concentration emulsions were made fresh.

Water-in-oil emulsions were prepared by the agent-in-oil method at room temperature ($\sim 25^{\circ}\text{C}$). The oil phase was the same as that used in the preparation of oil-in-water emulsions. 1.5% by volume (based on oil phase) of SPAN 80 (non-ionic emulsifier) was added to the oil phase and this mixture was stirred gently until the emulsifier dissolved completely. To this mixture, the required amount of deionized water was slowly added and the mixture was sheared in the Homo-mixer for about 15 minutes. To obtain higher concentration emulsions, more water was added and the mixture sheared again for 15 minutes.

Viscometers Used:

a) Rotating spindle in an infinite fluid:

The Brookfield Synchro-lectric viscometer falls under this category. The Brookfield model used in this study was provided with 4 spindles (one cylindrical and 3 disc type) and had 8 speeds (60 rpm to 0.3 rpm). It is one of the simplest types of instrument and is widely used throughout many industries. A synchronous motor drives a cylinder or disc element which is immersed in a fluid at a constant speed of rotation. The force required to overcome the resistance offered by the sample to this motion is derived from a beryllium copper spring which drives the immersed element. A dial at the top of the viscometer records the extent to which the spring is wound in overcoming the viscous resistance. Consequently the dial reading is proportional to the viscosity of the sample. The instrument can be calibrated with a known viscosity standard. For Newtonian fluids, this type of instrument is good for absolute measurements. For non-Newtonian fluids, this instrument is not so good for quantitative analysis. The viscosity of non-Newtonians will depend on the rate of shear at which they are measured and the geometry of the spindle makes it extremely difficult to calculate the shear rate. However, this instrument is still very useful for qualitative analysis. The shearing rate for a particular spindle (and a particular fluid) is proportional to the speed of rotation and the shear stress is proportional to the dial reading. Therefore, flow curves can simply be obtained by plotting dial readings against rpm.

For low viscosity Newtonian emulsions, a Brookfield adapter was used. The adapter consists of a precision cylindrical spindle rotating inside an accurately machined tube. Before collection of any experimental data, the adapter was calibrated with 8.7 cp (25°C) viscosity standard and the calibration constant was found to be the same as specified by the manufacturing company.

b) Coaxial cylinder viscometer

The FAN viscometer (coaxial type) model 35 having a gap width of 1.17 mm was used in this study. The outer cylinder (R-1 rotor) had a radius of 1.8415 cm. The inner cylinder (B-1 bob) had a radius of 1.7245 cm. The Bob height was 3.8 cm. Six test speeds of 3, 6, 100, 200, 300 and 600 rpm were available. The test fluid was contained in the annular space or shear gap between coaxial cylinders and the outer cylinder was rotated. The torque on the inner cylinder was measured and the torque as a function of angular velocity provided the relation between shear stress and shear rate.

In this type of viscometer, the variation of the rate of shear throughout the sample depends on the width of the annular gap between the two cylinders. If this width is kept small, the variation in shear rate can be made insignificant and the shear rate can be assumed constant throughout the sample. For relatively large gaps, the mean rate of shear and the mean shear stress per unit length of cylinder are calculated at the geometric mean radius ($\sqrt{R_1 R_2}$). These are given by:

$$\dot{\gamma} = 2R_1 R_2 \Omega / (R_2^2 - R_1^2) = K_2 \Omega \quad (1)$$

and

$$\tau = \frac{T}{2\pi R_1 R_2} = K_3 \text{ (D.R.)} \quad (2)$$

However, if R_1 and R_2 are not close the above equations do not hold. Krieger and Maron (9) have shown that the rate of shear at the surface of the inner cylinder is given by an infinite series which converges rapidly when the ratio of R_2/R_1 less than 1.2. For values of R_2/R_1 is less than 1.2 the expression for the shear rate at the inner cylinder reduces to

$$\dot{\gamma} = \frac{4\pi\eta\omega^2}{\beta^2 - 1} \left[1 + k_1 \frac{(1 - 1)}{s} + k_2 \frac{(1 - 1)^2}{s} \right] \quad (3)$$

where β , k_1 and k_2 are instrument constants given by

$$\beta = R_2/R_1 \quad (4)$$

$$k_1 = \frac{\beta^2 - 1}{2\beta^2} \left[1 + \frac{2}{3} \ln \beta \right] \quad (5)$$

$$k_2 = \frac{\beta^2 - 1}{6\beta^2} \ln \beta \quad (6)$$

and in any experiment s is the slope of the logarithmic plot of dial reading against rotational speed, ω , at a particular value of ω used in the experiment.

Hence shear stress and the corresponding rate of shear can be found from Eqns. (2) and (3) respectively and the flow curve then follows directly by repeating the experiment over a range of speeds.

The calibration of the instrument was done with a 100 cp viscosity standard. Since R_2/R_1 was less than 1.2, Eqn. 3 was used to calculate the shear rate. Shear stress was determined by multiplying the known viscosity with the shear rate. From known shear stress, K_3 was determined by using Eqn. 2. K_3 was found to be constant and was 0.5107 $\text{NM}^{-2}/\text{dial reading}$.

c) Cone-and-plate viscometer

The Weissenberg Rheogoniometer falls under this category. The model used in this study was R18. The platen diameter was 100 mm and cone angle was 4° , giving a clearance between the cone flat (truncated tip) and adjacent plate of $171 \mu\text{m}$. The flat bottom platen of the Rheogoniometer was replaced by a platen with disked end to facilitate the use of emulsions in the instrument. This helped to retain the test material in the cone and plate assembly and data at higher shear rates could be obtained without any loss of material which could otherwise have occurred. The disked platen was calibrated with standards of known viscosities and edge effects were determined.

In this type of instrument, the cone remains stationary and the bottom platen is made to rotate. Since the angle between the cone and the flat platen is very small (and therefore the average gap width is corresponding small) the whole sample will be subjected to a constant rate of shear.

The linear velocity at radius r is Ωr and the gap width at radius r is $r \tan \alpha$. Hence, the rate of shear at radius r is given by

$$\dot{\gamma}(r) = \frac{\Omega r}{r \tan \alpha} = \frac{\Omega}{\tan \alpha} \quad (7)$$

This means that the rate of shear is constant throughout the sample and is independent of r .

Further

$$\dot{\gamma}(r) = \Omega / \alpha \quad (8)$$

since α is small.

Also since $\dot{\gamma} = f(\tau)$ and $\dot{\gamma}$ is constant, then τ is constant.

Therefore,

$$T = 2\pi\tau \int_0^R r^2 dr \quad (9)$$

hence,

$$\tau = \frac{3T}{2\pi R^3} \quad (10)$$

where T is the total torque on the top platen (cone). The flow curve can thus be constructed by plotting the rate of shear ($\dot{\gamma}$) against the corresponding shear stress ($3T/2\pi R^3$).

Brookfield viscosity standards of 100 cp and 1000 cp were used to calibrate this instrument. For different speeds of rotation, shear rates were calculated according to Eqn. 8. Since shear rates were known and viscosity was also known, shear stresses were easily determined from

$$\tau = \mu \dot{\gamma} \quad (11)$$

Instrument calibration constant, K^1_T , was then determined from

$$\tau = K^1_T \Delta T t \dot{\gamma} \quad (12)$$

or

$$K^1_T = \frac{\tau}{\Delta T t \dot{\gamma}} \quad (13)$$

where ΔT is transducer reading in microns and $t = 2\pi/\Omega$, sec.rev.

The calibration constant, K^1_T , remained constant for both viscosity standards and its value was 6.72×10^{-4} NM⁻²/microns(sec/rev)sec⁻¹.

Photomicrographs

Photomicrographs of 70% oil-in-water emulsion were taken before and after shearing in the Weissenberg Rheogoniometer to see if the structure of the emulsion was altered during testing. The emulsion was sheared at a shear rate of 340.56 sec⁻¹ for 5 minutes.

A Zeiss optical microscope (with a camera attached) was used to take the photomicrographs. Emulsion was first diluted to about 20 times by glycerol and this diluted sample was then placed on a dimple slide. Transmitted light with green filter was used while taking photomicrographs.

RESULTS AND DISCUSSION

Oil-in-Water Emulsions Results

Figures 1-3 show the flow curves of oil-in-water emulsions obtained for various oil concentrations with different instruments. Since it was not possible to calculate shear rates directly in case of Brookfield viscometer, the data has been plotted as rpm versus the dial reading. The shape of the flow curves (characteristic of pseudoplastic/yield pseudoplastic fluids) obtained with different instruments are similar, however, an interesting point to note is that the Brookfield viscometer shows a definite yield stress for concentrated emulsions ($\phi \geq 76\%$) whereas the Weissenberg Rheogoniometer does not show any yield stress. The Fan viscometer also appears to show a yield stress for concentrated emulsions.

In Figure 4, the data of shear stress (τ) versus the shear rate ($\dot{\gamma}$) has been plotted on a log-log scale. The dotted line for the 30% emulsion indicates a viscosity measurement from the Brookfield adapter. The other lines shown on the figure are drawn through the Weissenberg Rheogoniometer data. Up to 60% concentration, there is a good agreement between the instruments. For 70% emulsion, there is a satisfactory agreement between the Fan viscometer and Weissenberg Rheogoniometer although there is some scattering of data at the lower shear rates. This scattering could be due to reasons other than slip effects. For oil concentrations greater than 70% (i.e. for 76%, 80% and 82% emulsions), significant deviation is seen between the Weissenberg Rheogoniometer and the Fan viscometer data. The Rheogoniometer gives lower viscosities than the Fan viscometer at the same shear rate for the same emulsion. In Figure 5, the shear rate versus shear stress data has been replotted on semi-log plot for oil concentrations greater than 70%. A nearly parallel shift in flow curves obtained from the Fan viscometer and the Rheogoniometer is obvious. For the 70% emulsion, a similar flow curve is obtained.

Water-in-oil Emulsion Results

Figures 6-8 show the flow curves of water-in-oil emulsions obtained for various water concentrations with different instruments. The Brookfield and Fan viscometers exhibit yield stress for concentrated emulsions ($\phi > 60\%$) whereas the Weissenberg Rheogoniometer does not show any yield stress. Figure 9 shows the rheograms (τ vs. $\dot{\gamma}$) obtained with different instruments on log-log scale. The dotted lines for 0%, 10% and 20% emulsions indicate viscosity measurements from the Brookfield adapter. The other lines shown on the figure are drawn through the Weissenberg Rheogoniometer data. Up to 60% concentration, there is excellent agreement between the instruments. However, for water concentrations greater than 60%, flow curves obtained by different instruments are appreciably

different for the same concentration emulsions. As in the oil-in-water emulsions, the Rheogoniometer gives lower viscosities than Fan viscometer at the same shear rate. Figure 10 shows that there is a nearly parallel shift in the flow curves obtained from the two instruments. For 60% emulsion, a single flow curve is obtained.

Discussion

The shift in the flow curves obtained from the different instruments for the same emulsions can be explained by slip effects in the Weissenberg Rheogoniometer. Slip effects are known to exist in capillary tubes and small diameter pipes (10-15).

When a dispersed system enters a narrow capillary or tube, the dispersed phase shows a tendency to migrate away from the tube wall towards the tube axis. If this occurs there is a concentration fluctuation across the capillary with the major reduction in concentration in the layers adjacent to the capillary wall.

This phenomenon has actually been observed with a suspension of paper pulp (16). The fluid in the region close to the wall has a lower viscosity and this gives rise to an effective velocity of slip at the wall. Different diameter capillaries will then give different flow curves. Oldroyd (17) has done a detailed analysis of slip effects in pipes.

It is highly likely that slip effects would also exist in cone-and-plate type viscometers. Due to preferred orientation of oil particles, a region adjacent to the cone and plate may form which is deficient in dispersed material. This will lead to a slip effect at the cone and plate surface resulting in lower viscosity measurements. Boardman and Whitmore (18) have also observed similar effects for flocculated china clay suspensions. These suspensions are normally Bingham plastic in nature. However, when these

suspensions are studied in a cone and plate viscometer, they appear to behave as pseudo plastic fluids (with no yield stress) and the linear portion of the flow curve (shear stress vs. shear rate) is well below the true flow curve. The authors have attributed this abnormal behaviour to the presence of effective slip at the cone and platen surface.

It is highly likely that concentrated oil-in-water ($\phi \geq 76\%$) and water-in-oil ($\phi > 60\%$) emulsions exhibit some real yield stress (as shown by the Fan and Brookfield viscometers). Due to abnormal wall effects at the boundaries, they do not show this yield stress when the data is obtained from cone-and-plate type viscometer.

Estimation of Continuous Phase Film Thickness

One can estimate the thickness of a continuous phase film formed adjacent to a cone and plate by theoretical analysis. Figure 11 shows a schematic of the cone-and-plate viscometer. The cone tip is truncated in order to avoid disturbance of torque readings although for the sake of accuracy, the point of the cone should touch the centre of the flat plate. Truncation of cone tip would give only negligible error since the torque produced at the centre of the specimen is immeasurably small. The gap width (between truncated tip and flat plate) is so adjusted that the imaginary tip of the cone would touch the centre of the plate.

If "slip effects" are absent, then:

$$\text{shear stress } \tau = \frac{3}{2\pi R^3} T \quad (14)$$

and shear rate is given by Eqn. (8).

where T is torque, Ω is angular velocity and α is cone angle. Therefore, by measuring the torque on the cone over a range of angular velocities Ω the flow curve of the fluid is obtained. For yield pseudoplastic fluid,

$$\tau = \tau_0 + K \gamma^n \quad (15)$$

where τ_0 is yield stress of the fluid and K and n are constants.

If "slip effects" are present, inhomogeneity development in the fluid adjacent to the walls, produces a layer of continuous phase film which possesses different flow properties from the bulk fluid. τ and γ (as given by Eqns. 9 & 8) are then only apparent values. In our analysis, we will make the following assumptions regarding the nature of the film fluid. Boardman and Whitmore (18) and Reiner (19) made similar assumptions when calculating wall effects in the case of Bingham plastic fluids.

- (i) It is Newtonian in nature.
- (ii) Its thickness is constant and is independent of the thickness of the bulk fluid.
- (iii) Its thickness is independent of the rate of shear.
- (iv) The liquid abstracted from the bulk fluid to form the wall layer is sufficiently small to produce a negligible change in the properties of the bulk fluid.

Now let the sheared volume of a cone-and-plate viscometer be completely filled with a yield pseudoplastic fluid except at the walls where it is bounded by a layer of liquid of thickness d and viscosity μ_w .

Then consider a narrow annulus of fluid situated at radius r . Because the annulus is narrow, dr , can be regarded as constant within it so that conditions in the annulus are equivalent to those between two parallel plates situated

distance αr apart. The shear stress-shear rate relationship of a yield pseudoplastic fluid in such an annulus can very easily be shown to be

$$\tau = \tau_0 + K \left[\frac{\frac{\Omega r}{\alpha r} - \frac{2\tau_0 d}{\mu_w}}{2d} \right]^n \quad (16)$$

Thus τ changes with r and in general there are two regions:

- (i) when $\tau < \tau_0$ the yield pseudoplastic fluid is unsheared, all relative motion occurring in the wall layers. This will be termed "lubricated plug flow". (Boardman and Whitmore (1963)).
- (ii) when $\tau > \tau_0$ the yield pseudoplastic fluid and the wall layers are all fully sheared. This will be termed "complete laminar flow".

If the radius of transition from lubricated plug flow to complete laminar flow = ℓ_2 , Eqn. 15 gives

$$\tau_0 = \tau_0 + K \left[\frac{\frac{\Omega \ell_2}{2\ell_2} - \frac{2\tau_0 d}{\mu_w}}{2\ell_2 - 2d} \right]^n$$

or

$$\ell_2 = \frac{2\tau_0 d}{\Omega \mu_w} \quad (17)$$

if $\ell_2 > R$ complete laminar flow is absent from the viscometer. From Eqn. 16 the condition is given by

$$\Omega = \frac{2\tau_0 d}{\mu_w R}$$

or

$$\gamma_1 = \frac{2\tau_0 d}{\alpha \mu_w R} \quad (18)$$

where γ_1 is the maximum apparent rate of shear without any complete laminar flow. Therefore, only lubricated plug flow exists for shear rates: $0 < \gamma < \gamma_1$.

For lubricated plug flow

It can be very easily shown that

$$\tau = \left(\frac{\mu_w \alpha}{2d} \right) (\dot{\gamma}) r \quad (19)$$

$$T = \int_0^R 2\pi r^2 \tau dr \quad (20)$$

or

$$T = 2\pi \left(\frac{\mu_w \alpha}{2d} \right) \gamma \left(\frac{R^4}{4} \right) \quad (21)$$

$$\tau = \frac{3}{2\pi R^3} \left[2\pi \left(\frac{\mu_w \alpha}{2d} \right) \gamma \left(\frac{R^4}{4} \right) \right]$$

or

$$\tau = \left(\frac{3 \mu_w \alpha R}{8d} \right) \gamma \quad (22)$$

Thus the apparent flow curve (determined from cone-and-plate viscometer) of τ against r is linear. From the slope of this curve, one can determine d , the thickness of the film. Table I summarizes the values of film thickness obtained for various concentrated emulsions. It appears from the calculations (given in Table I) that the film thickness decreases as the concentration of dispersed phase in the emulsions is increased.

Table 1. Continuous phase film thickness

Type of Emulsion	dispersed phase concentration	slope of initial portion of τ vs γ curve	yield stress NM^{-2}	film thickness, μm
water-in-oil	70%	21	5	0.126
water-in-oil	75%	22.44	18	0.118
oil-in-water	76%	6.0	4	0.226
oil-in-water	80%	7.75	7	0.176
oil-in-water	82%	27.5	10	0.05

Variation of Viscosity with the Dispersed Phase Concentration

The viscosity of emulsion depends upon several factors:

1. volume concentration of dispersed phase
2. viscosity of continuous phase
3. size and distribution of dispersed particles
4. temperature
5. shear rate

Since in this study rheological data has been collected at constant temperature (room temperature), temperature is not a variable. Also, preliminary experiments indicated that size and distribution of dispersed particles did not vary significantly with the dispersed phase concentration for constant speed of stirring. The viscosity of continuous phase was also constant. Therefore, there are only two variables to be considered: dispersed phase concentration and shear rate.

Richardson's equation (20) is very often used to correlate the data of emulsion viscosity (21-22):

$$\nu_r = \exp (K_1 \phi) \quad (23)$$

where ν_r is the relative viscosity of emulsion (which is defined as the ratio of emulsion to external phase viscosity), ϕ is the dispersed phase concentration and k_1 is a constant. However, it is necessary to point out that this equation is valid only for Newtonian emulsions and emulsions behave like Newtonian fluids only at low (usually less than 30%) dispersed phase concentrations. At higher dispersed concentrations, viscosity of emulsions will also depend on shear rate. Figure 12 shows the plot of relative viscosity (at a shear rate of 200 s^{-1}) and plastic viscosity, which is the slope of the linear portion of stress vs. shear rate curve, against dispersed phase concentration. The data plotted in Figure 12 is obtained from the Fan viscometer and is for oil-in-water emulsions. It is obvious from the figure that Richardson's equation is not valid for concentrated emulsions. According to Richardson's equation ν_r versus ϕ should be linear on a semi-log plot. A linear relationship exists only at lower concentrations ($\phi \leq 30\%$). The viscosity of emulsions is higher at lower shear rates indicating that emulsions are shear-thinning.

Figure 13 shows a plot similar to Figure 12 but for water-in-oil emulsions. As for the oil-in-water emulsion Richardson's equation again fails to correlate higher concentration data. The relationship between relative viscosity and ϕ is linear only for ϕ less than 25%. Water-in-oil emulsions are also shear-thinning as shown by Figure 13.

Effect of Shearing in the Rheogoniometer on the Structure of the Emulsion

In Figure 14, photomicrographs of 70% oil-in-water emulsion before and after shearing (shearing done for 5 minutes at 340.56 sec^{-1}) are presented. On comparison of the photomicrographs, it is evident that particle size and distribution was not significantly altered during rheological testing in the Rheogoniometer.

CONCLUSIONS

The main conclusions of this study are:

1. Up to 70% oil concentration, oil-in-water emulsions do not exhibit abnormal wall effects. The flow curves obtained from coaxial cylinder and cone-and-plate viscometers are essentially the same. At concentrations greater than 70%, "slip effects" are present in the Weissenberg Rheogoniometer. The Rheogoniometer gives appreciably lower viscosities than the Fan viscometer at the same shear rate (for the same emulsions) and this is probably due to the slip effects at the walls of the cone and plate viscometer.
2. Up to 60% water concentration, water-in-oil emulsions do not exhibit "slip effects". The flow curves obtained from Fan viscometer and Weissenberg Rheogoniometer are the same. At concentrations greater than 60%, "slip effects" are present in the Weissenberg Rheogoniometer and the viscosities determined by Rheogoniometer are much lower than those given by the Fan viscometer.
3. Flow curves obtained from Fan and Brookfield viscometers exhibit yield stress for concentrated oil-in-water and water-in-oil emulsions. However, the cone-and-plate viscometer does not exhibit yield stress. This also supports the slip effect theory.
4. Higher concentration emulsions (both oil-in-water and water-in-oil) do not follow Richardson's equation even for constant rate of shearing. Curvature in the plot of $\log u_r$ against $\dot{\gamma}$ is seen at higher $\dot{\gamma}$. Thus the emulsions are non-Newtonian in nature.
5. A method of estimation of continuous phase film thickness (which leads to "slip effects") has been developed. The film thickness decreases with increased concentration of emulsion.

6. Photomicrographs of 70% oil-in-water emulsion before and after shearing in the Rheogoniometer (at 340.56 s^{-1}) revealed that particle size and distribution of emulsion was not significantly altered during testing in the Rheogoniometer.

ACKNOWLEDGEMENTS

Appreciation is expressed to CANMET for a research contract to enable this investigation to proceed.

NOMENCLATURE

d_0	gap width between truncated cone tip and flat platen
d	thickness of the continuous phase film formed adjacent to cone and plate surface
D.R.	dial reading
k_1	instrument constant defined by Eqn. 5
k_2	instrument constant defined by Eqn. 6
K	consistency index, Eqn. 14
K_1	constant in Eqn. 22
K_2	constant in Eqn. 1
K_3	constant in Eqn. 2
L_2	radius of transition from lubricated plug flow to complete laminar flow
n	flow behaviour index, Eqn. 14
r	radial distance
R	radius of cone
R_1	radius of inner cylinder (bob) of coaxial cylinder viscometer
R_2	radius of water cylinder (rotor) of coaxial cylinder viscometer
T	torque

Greek Letters

α	cone angle
R	R_2/R_1 , ratio of radius of outer cylinder to inner cylinder of coaxial cylinder viscometer
ΔT	transducer reading
ϕ	volume concentration of dispersed phase
γ	shear rate
$\bar{\gamma}$	mean shear
γ_1	maximum rate of shear without any complete laminar flow
μ	viscosity
μ_r	relative viscosity
μ_w	viscosity of continuous phase film formed adjacent to cone and plate surface
Ω	angular velocity
τ	shear stress
τ_0	yield stress
$\bar{\tau}$	mean shear stress

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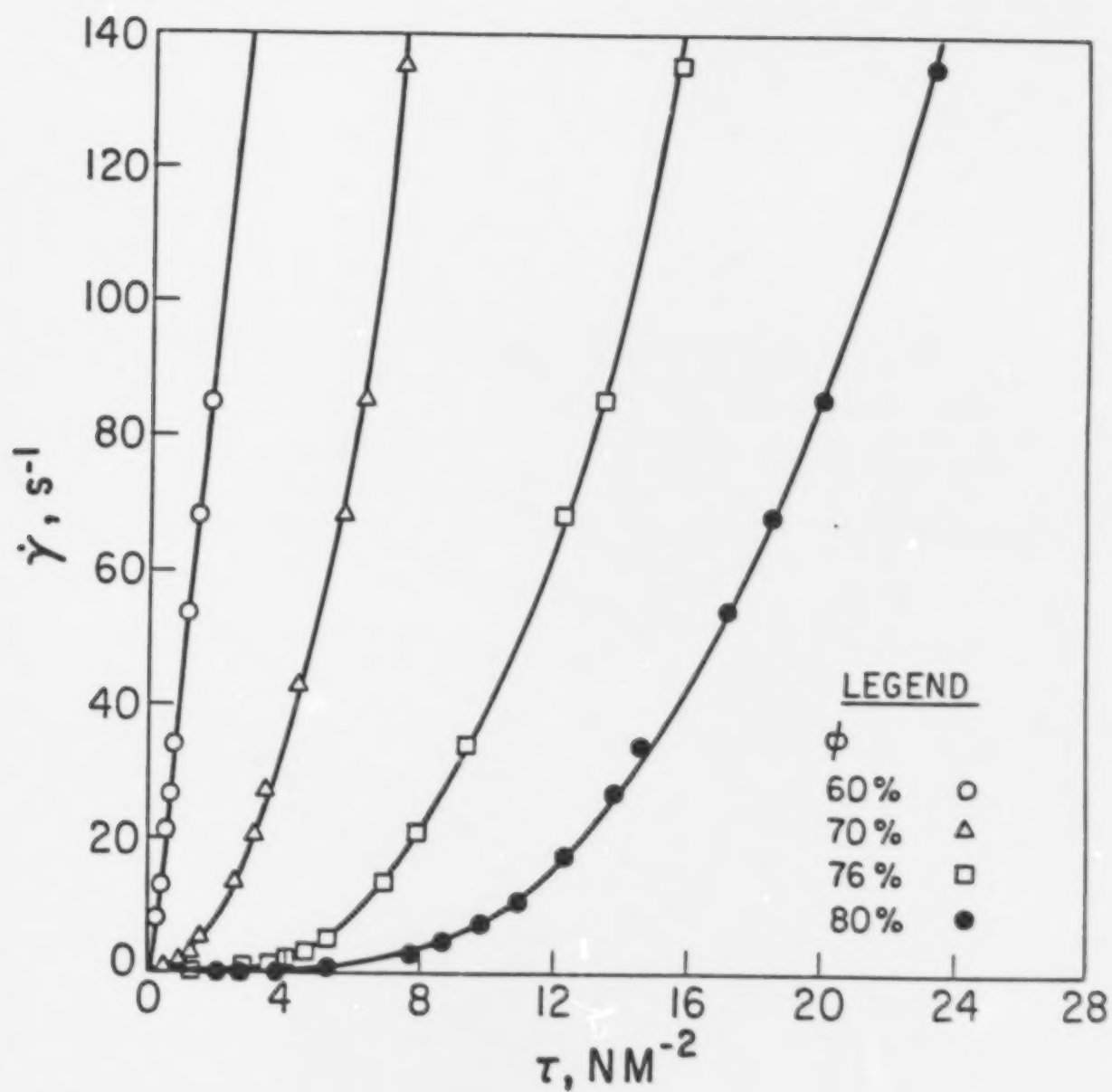


Figure 1. Flow curves of oil-in-water emulsions obtained by a Weissenberg Rheogoniometer.

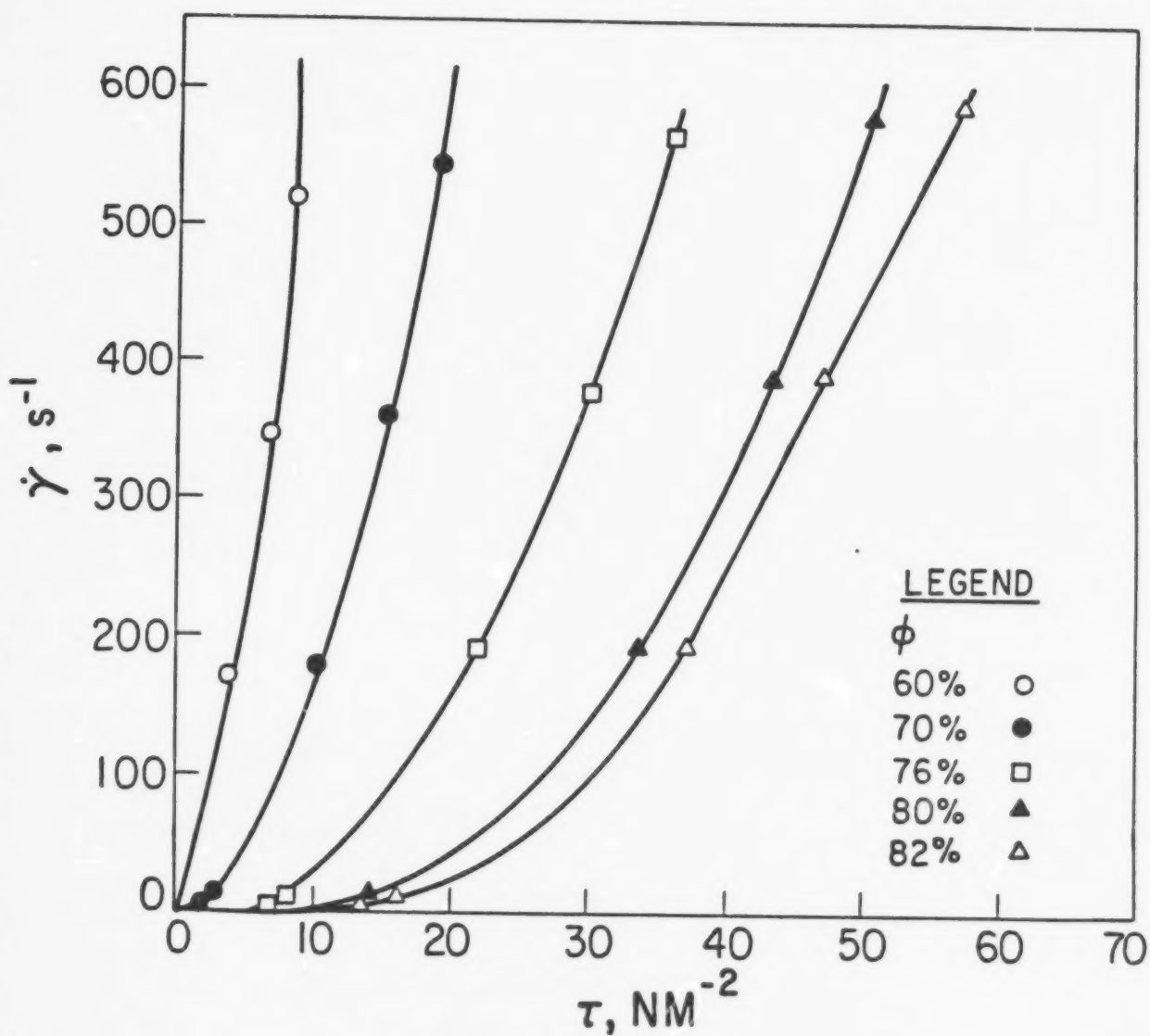


Figure 2. Flow curves of oil-in-water emulsions obtained by a Fann Viscometer.

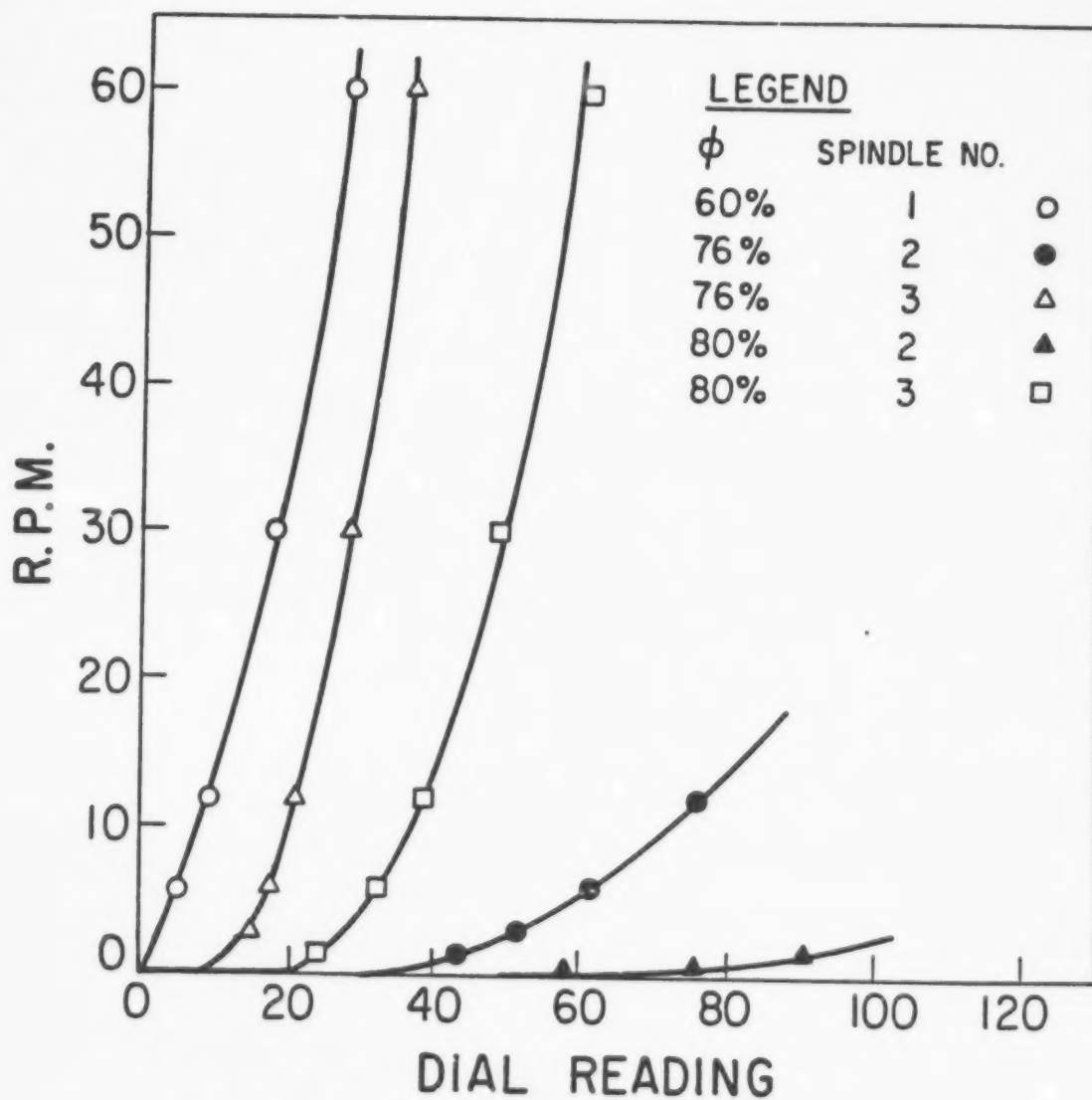


Figure 3. Flow curves of oil-in-water emulsions obtained by the Brookfield viscometer.

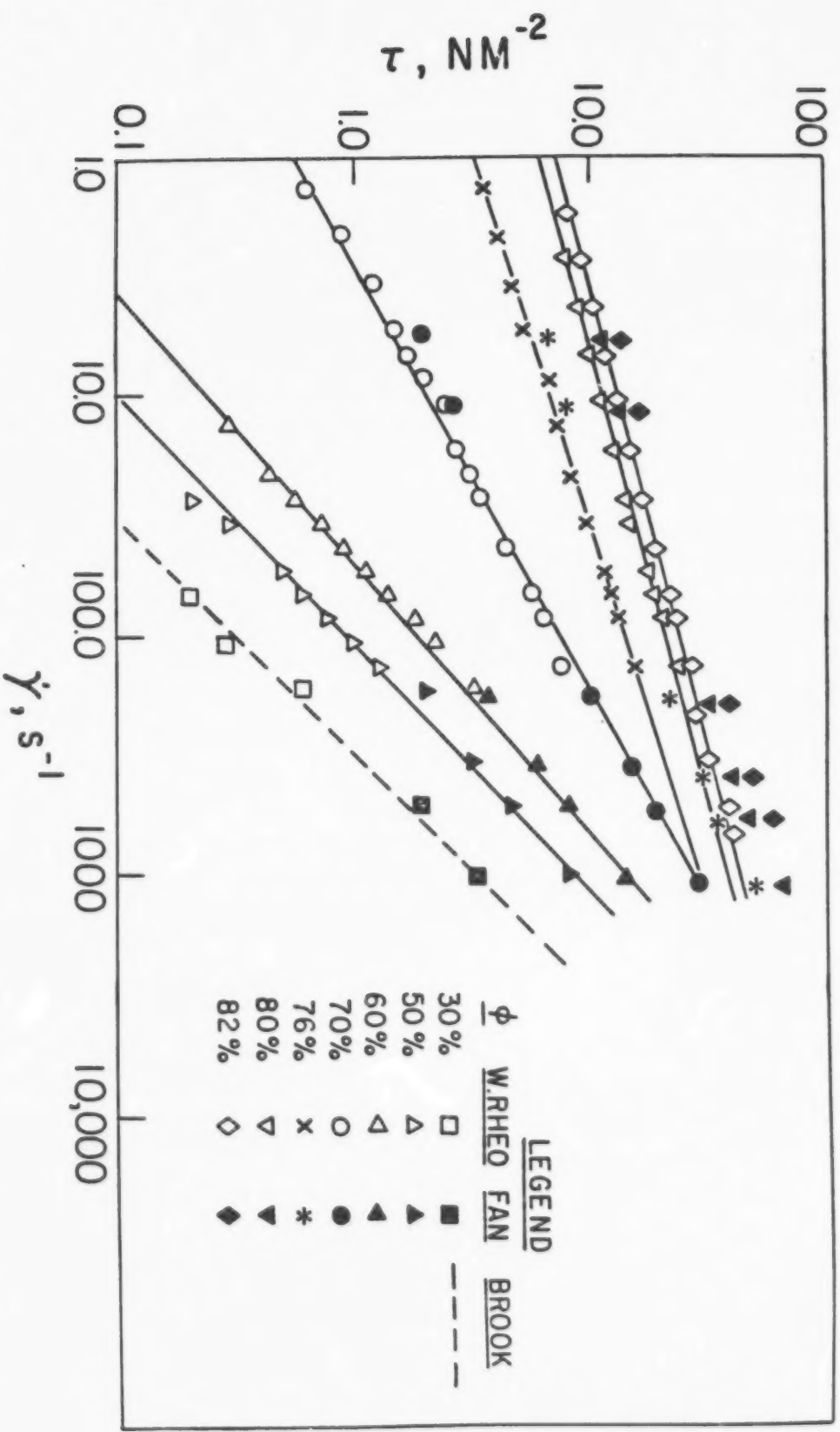


Figure 4. Comparison of flow curves obtained from different instruments for oil-in-water emulsions.

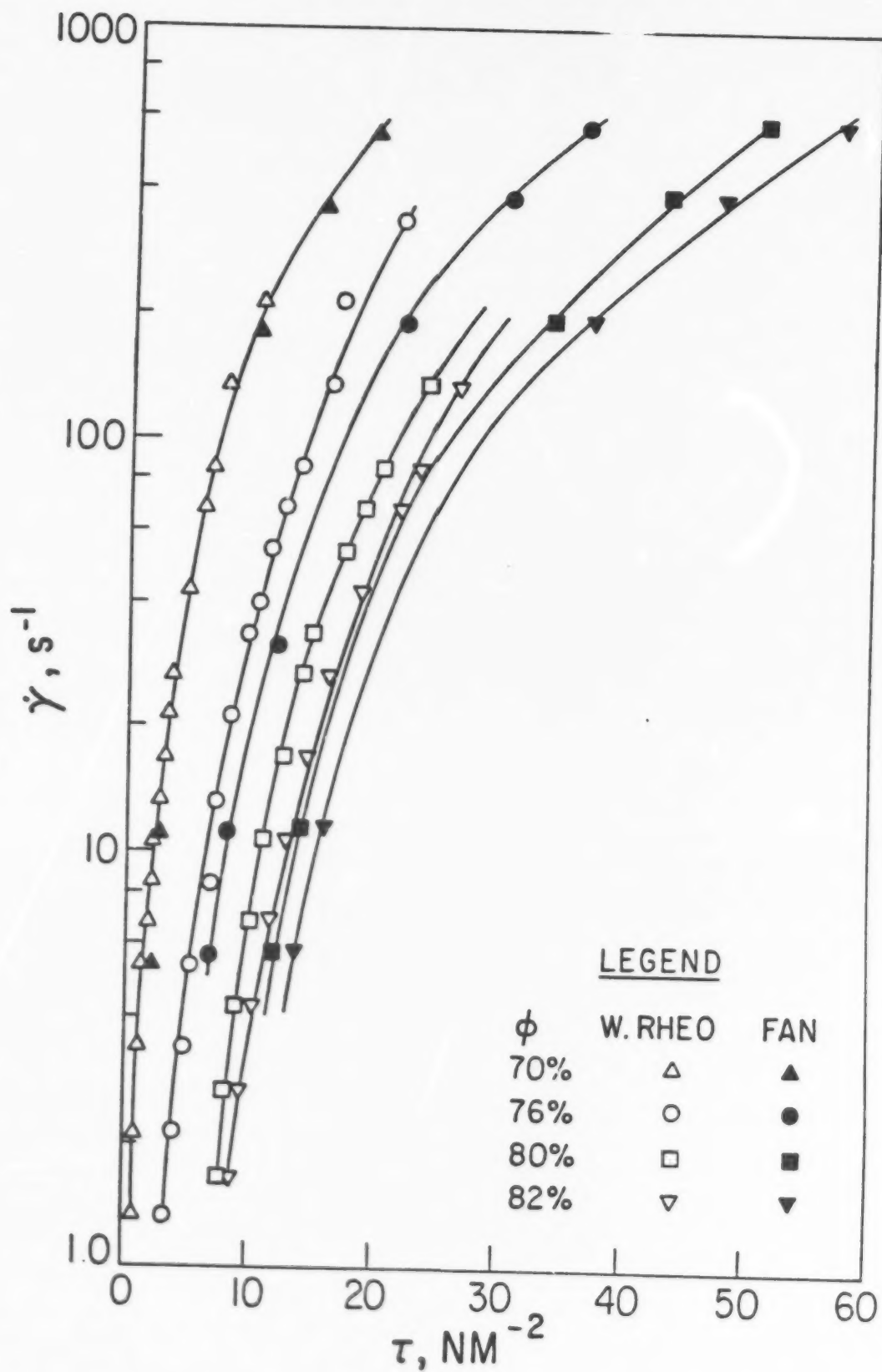


Figure 5. Comparison of higher concentration oil-in-water emulsion data obtained from different instruments.

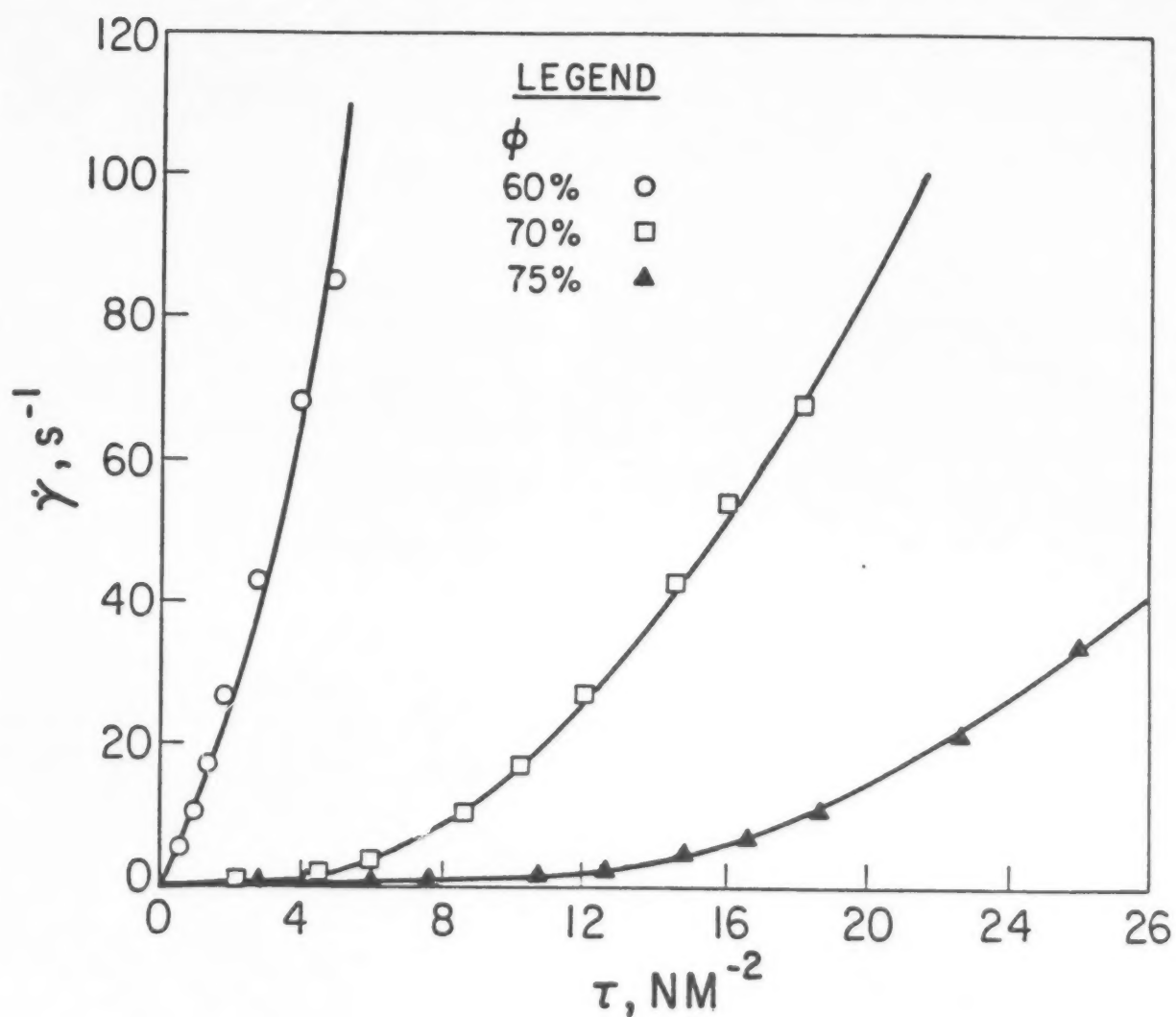


Figure 6. Flow curves of water-in-oil emulsions obtained by a Weissenberg Rheogoniometer.

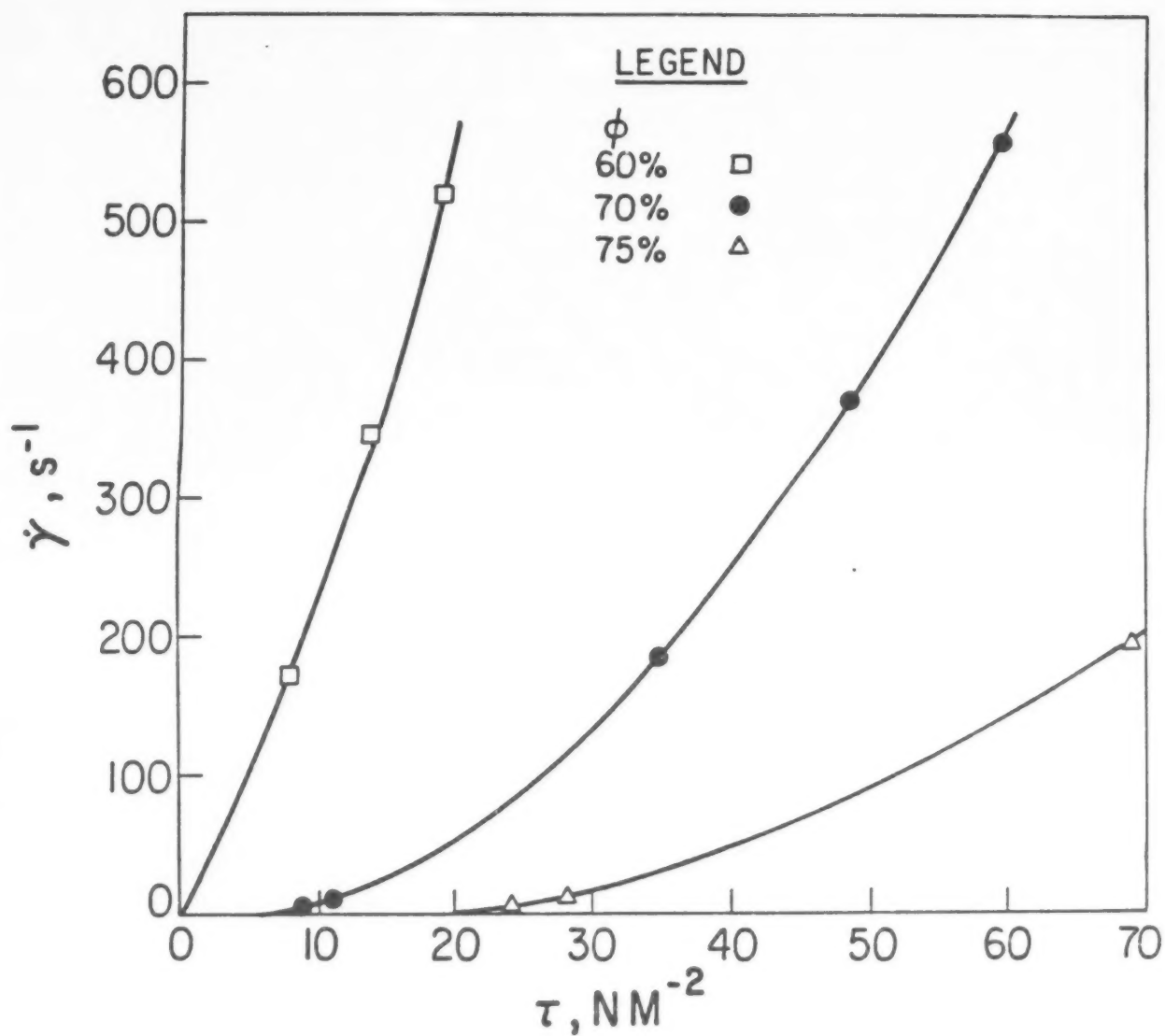


Figure 7. Flow curves of water-in-oil emulsions obtained by a Fann Viscometer.

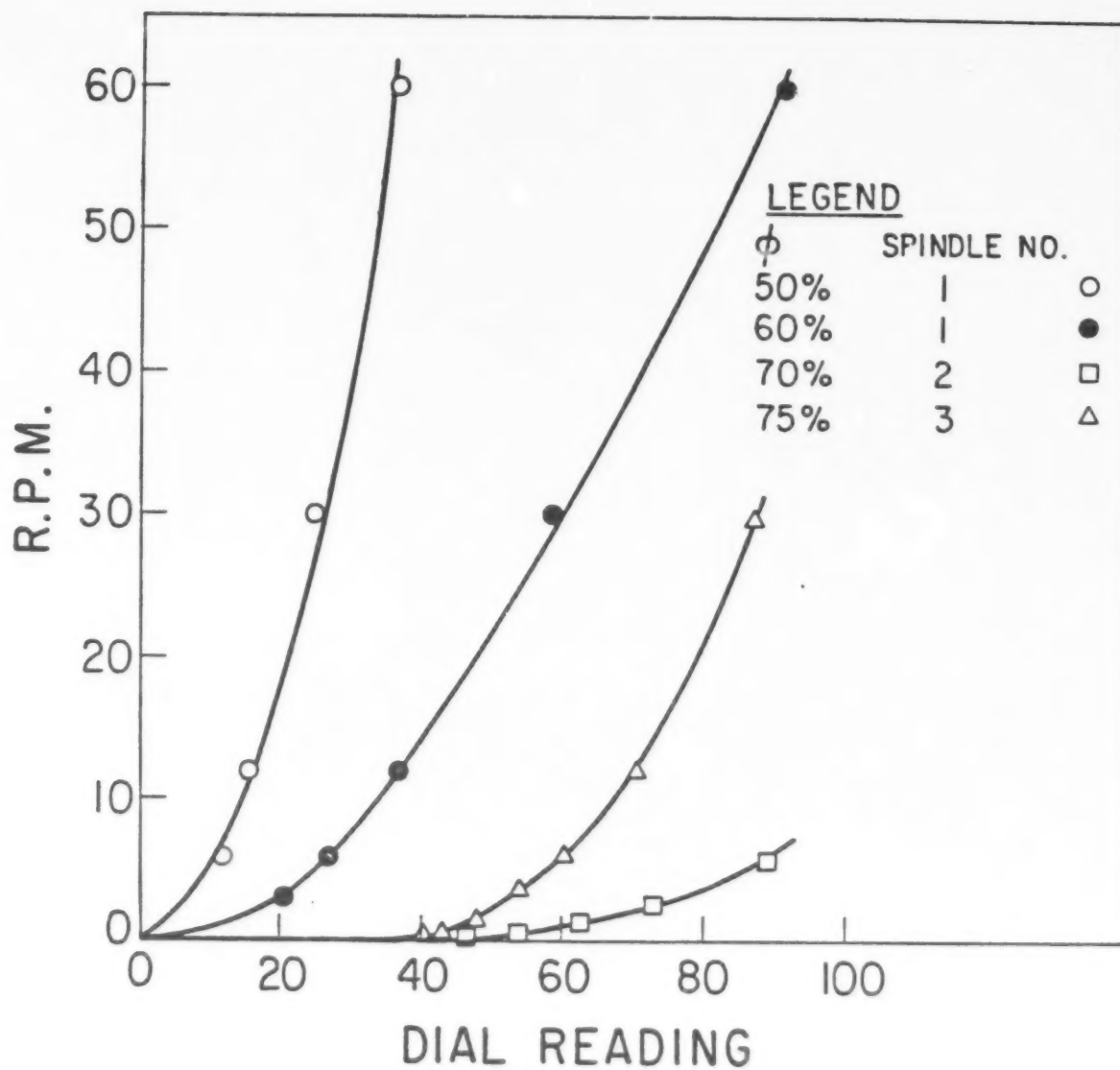


Figure 8. Flow curves of water-in-oil emulsions obtained by a Brookfield viscometer.

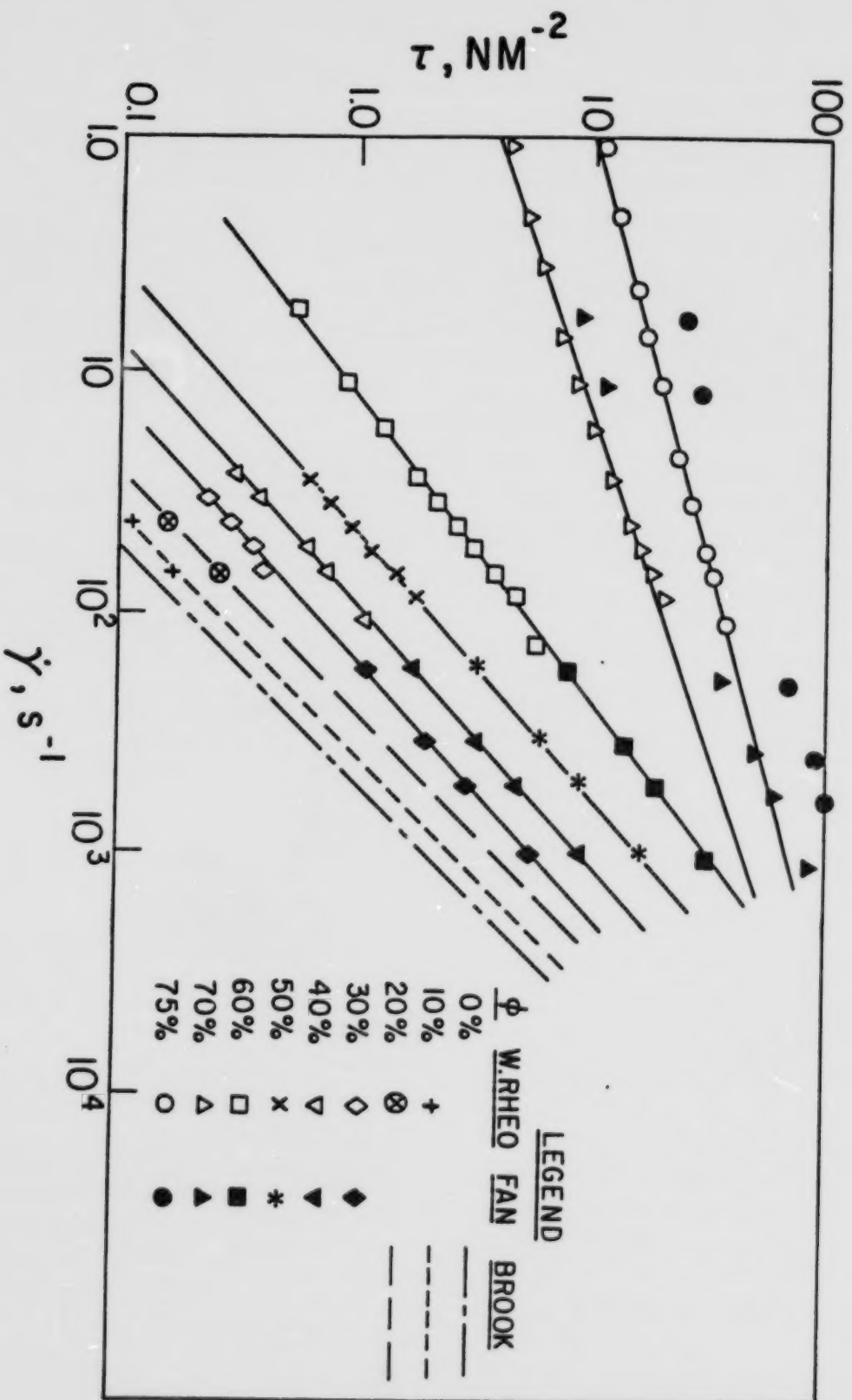


Figure 9. Comparison of flow curves of water-in-oil emulsions obtained from different instruments.

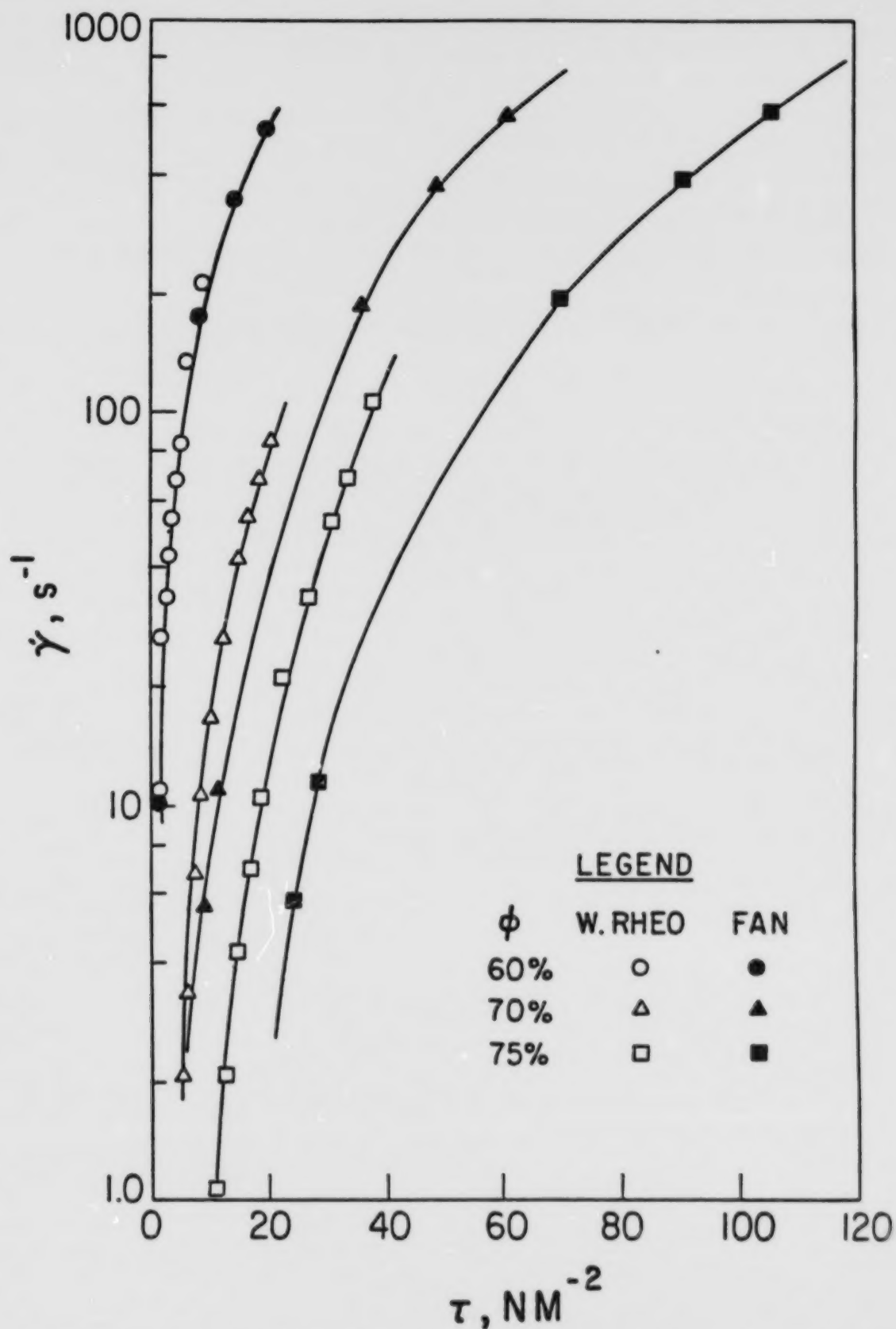


Figure 10. Comparison of higher concentration water-in-oil emulsions data obtained from W. RHEO and FAN.

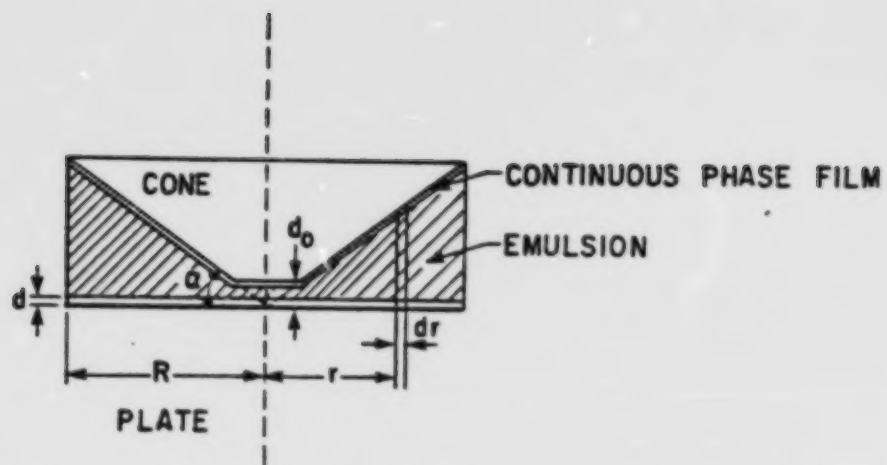


Figure 11. Schematic of Weissenberg Rheogoniometer.

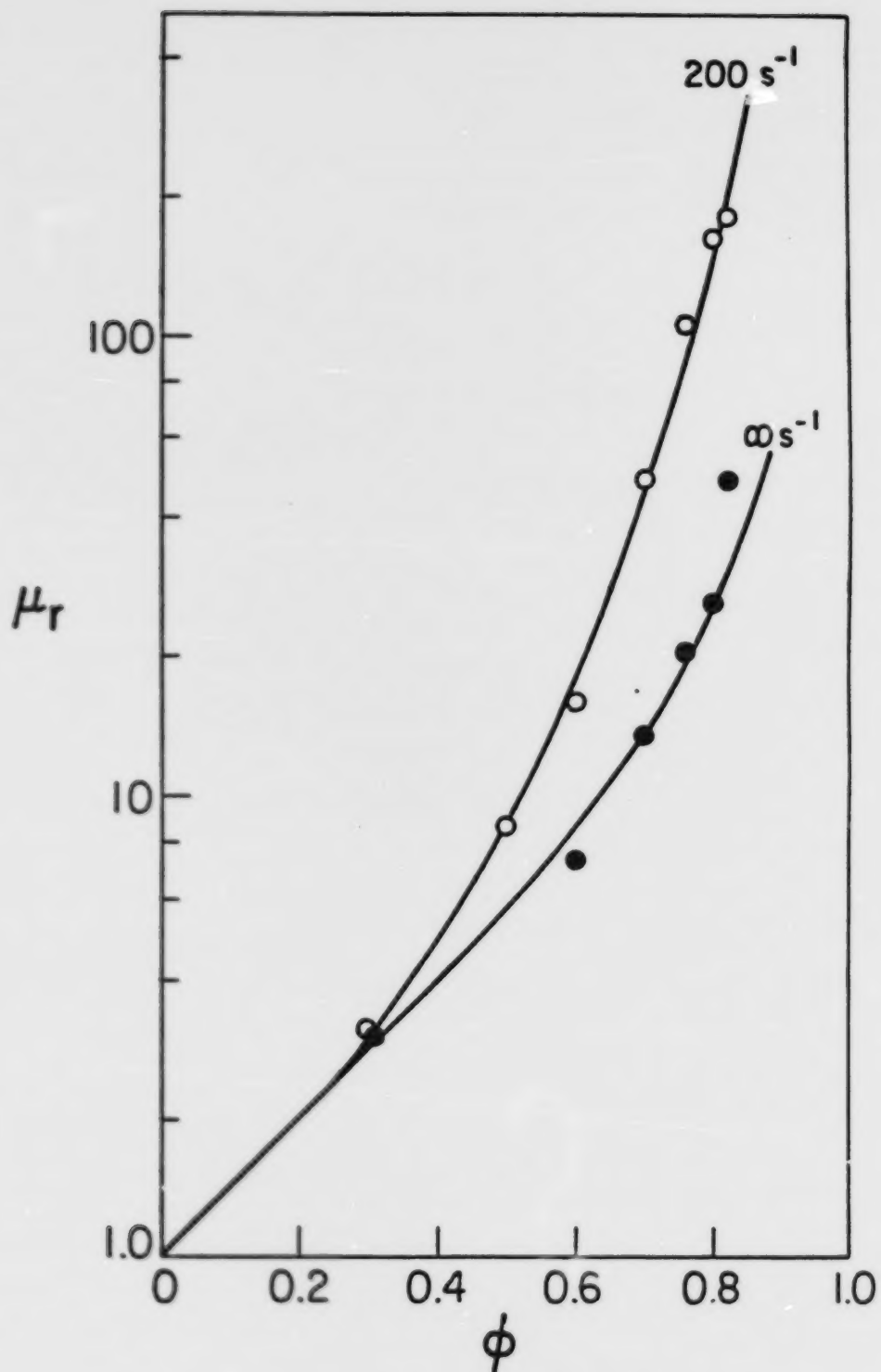


Figure 12. Relative viscosity versus the oil concentration for oil-in-water emulsions.

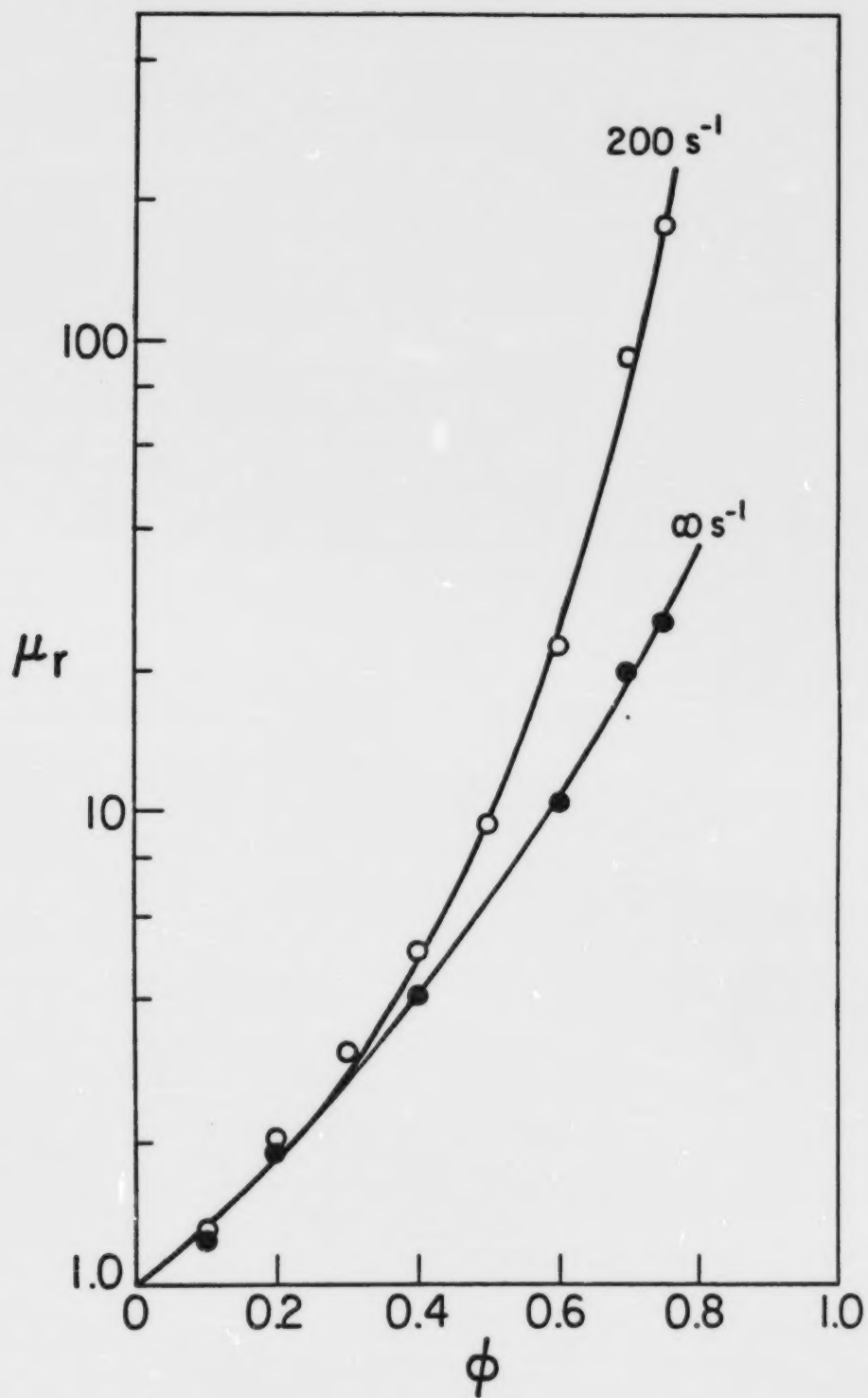
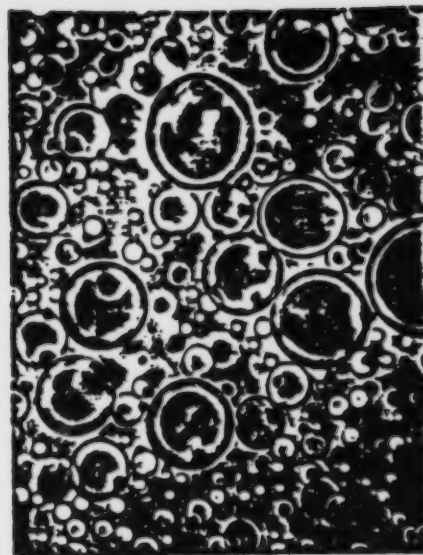
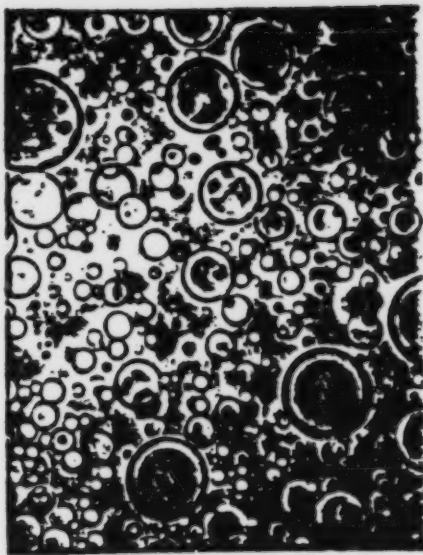
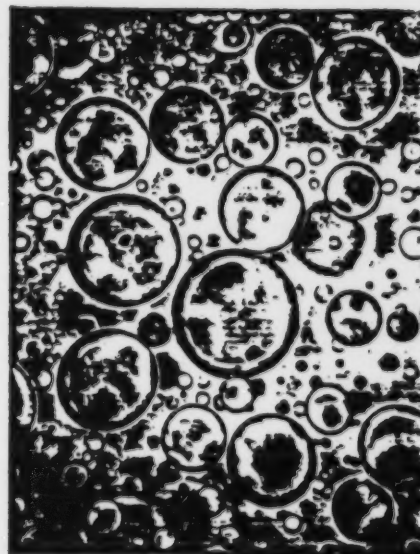
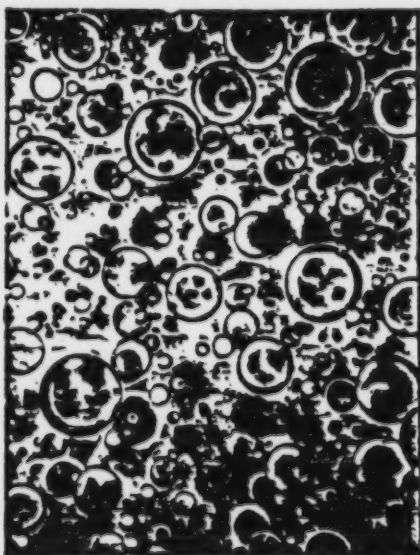


Figure 13. Relative viscosity versus the water concentration for water-in-oil emulsions.



Photomicrographs before Shearing



Photomicrographs after Shearing

Fig. 14 Photomicrographs of 70% o/w emulsion before and after shearing

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ABSTRACT

In this study, the rheological data obtained for various concentrated oil-in-water and water-in-oil emulsions from viscometers of different design and geometry has been analyzed and compared. The three types of viscometers used are: coaxial cylinder, cone-and-plate and rotating bob in an infinite fluid.

In the case of oil-in-water emulsions, the flow curves obtained from the cone-and-plate viscometer are in close agreement with those given by coaxial cylinder viscometer only when the oil concentrations are below 70%. For concentrations greater than 70%, the flow curves obtained from the two instruments are appreciably different.

In the case of water-in-oil emulsions, the flow curves obtained from the different instruments are in agreement only when the water phase concentrations are below 60%. For water concentrations greater than 60%, the flow curves obtained are significantly different.

The variation in the flow curves for the same concentration emulsions has been attributed to the presence of "slip-effects" in the cone-and-plate viscometer.

INTRODUCTION

A considerable amount of the world's crude oil is produced in the form of stable emulsions (1). The knowledge of accurate rheological properties of emulsions is very important for understanding their flow behaviour in porous media and for the design of pipelines and pumping equipment etc.

To ensure that the rheological properties determined are the properties of emulsion and not artifacts produced by the method of investigation, it is very important and often necessary to obtain comparative data from viscometers of different designs. The main objective of this study was to analyze and compare emulsion rheological data obtained from instruments of different design and determine if "slip effects" were present.

EXPERIMENTAL

Emulsion Preparation:

Oil-in-water emulsions were prepared by the agent-in-water method at room temperature ($\sim 24^{\circ}\text{C}$). The oil phase was 73.3% by volume white mineral oil dissolved in 26.7% by volume carbon-tetrachloride which was added to the mineral oil in order to make the density of oil phase equal to that of water phase. In this way the creaming effect in emulsions was eliminated. One percent by volume (based on the water phase) of non-ionic emulsifier Triton x-100 was used to prepare the emulsions. The mixture of oil and water was sheared in a high shear machine (Gifford-Wood Model 1-LV Homogenizer-Mixer) for about 15 minutes.

Water-in-oil emulsions were prepared by the agent-in-oil method at room temperature ($\sim 25^{\circ}\text{C}$). The oil phase was the same as that used in the preparation of oil-in-water emulsions. 1.5% by volume (based on oil phase) of non-ionic emulsifier SPAN 80 was used to prepare the emulsions. High speed shearing was done in a homogenizer for about 15 minutes.

Viscometers Used:

a) Rotating spindle in an infinite fluid: The Brookfield Synchro-lectric viscometer falls under this category. The Brookfield model used in this study was LV with 4 spindles (one cylindrical and 3 disc type spindles) and having 8 speeds (60 rpm to 0.3 rpm). For Newtonian fluids, this type of instrument is good for absolute measurements. For non-Newtonian fluids, this instrument is not so good for quantitative analysis. The viscosity of non-Newtonians will depend on the rate of shear at which they are measured and the geometry of the spindle makes it extremely difficult to calculate the shear rate. However, this instrument is still very useful for qualitative analysis. The shearing rate for a particular spindle (and a particular fluid) is proportional to the speed of rotation and the shear stress is proportional to the dial reading. Therefore, flow curves similar to the true flow curves can be simply obtained by plotting dial reading against r.p.m.

For low viscosity Newtonian emulsions, a Brookfield U.L. adapter was used. The U.L. adapter consists of a precision cylindrical spindle rotating inside an accurately machined tube.

b) Coaxial cylinder viscometer: The Fann viscometer falls in the category of coaxial cylinder viscometers. Fann viscometer model 35 having gap width of 1.17 mm was used in this study. The outer cylinder (R-1 rotor) had a radius of 1.8415 cm and a 303 S.S. construction. The inner cylinder (B-1 bob) had a radius of 1.7245 cm and a 303 S.S. construction. The Bob height was 3.8 cm. Six test speeds of 3, 6, 100, 200, 300 and 600 r.p.m. were available.

For this type of instrument, the shear stress at the surface of inner cylinder is given by

$$\tau = K_1 (\text{Dial reading}) \quad (1)$$

For values of R_2/R_1 less than 1.2, the rate of shear at the inner cylinder is given by (2):

$$\frac{4\pi\Omega R^2}{\delta^2 - 1} [1 + k_1 \left(\frac{1}{s} - 1\right) + k_2 \left(\frac{1}{s} - 1\right)^2] \quad (2)$$

where δ , k_1 and k_2 are instrument constants given by

$$\delta = R_2/R_1 \quad (3)$$

$$k_1 = \frac{\delta^2 - 1}{2\delta^2} [1 + \frac{2}{3} \ln \delta] \quad (4)$$

$$k_2 = \frac{\delta^2 - 1}{6\delta^2} \ln \delta \quad (5)$$

and in any experiment s is the slope of the logarithmic plot of dial reading against rotational speed, Ω , at a particular value of Ω used in the experiment.

Hence shear stress and the corresponding rate of shear can be found from Eqs. (1) and (2) respectively and the flow curve then follows directly by repeating the experiment over a range of speeds.

c) Cone-and-plate viscometer: The Weissenberg Rheogoniometer falls under this category. The model used in this study was R 18. The platen diameter was 100 mm and cone angle was 4° , giving a clearance between the cone flat (truncated tip) and adjacent plate of 171 μm . Since the angle between the cone (stationary part) and the flat platen (rotating part) is very small, the whole sample is subjected to a constant rate of shear. The rate of shear is given by:

$$\dot{\gamma} = \Omega/\alpha \quad (6)$$

The torque on the top platen (cone) is given by:

$$T = 2\pi r \int \gamma^2 dy \quad (7)$$

and shear stress is given by:

$$\tau = \frac{3T}{2\pi R^3} = K_T \Delta T t \dot{\gamma} \quad (8)$$

The flow curve could be constructed by plotting the shear stress ($3T/2\pi R^3$) against the rate of shear (Ω/α).

Calibration of the Instruments: Before collection of any experimental data, the instruments were calibrated with the Brookfield viscosity standards of 8.7 cp, 100 cp and 1000 cp (at 25°C). K_1 , constant in Eq. 1, was found to be $0.5107 \text{ NM}^{-2}/\text{dial reading}$. K_T , constant in Eq. 8, was found to be $6.72 \times 10^{-2} \text{ NM}^{-2}/\text{microns (sec/rev) sec}^{-1}$.

RESULTS AND DISCUSSION

Figure 1 shows the rheograms of oil-in-water emulsions obtained from the different instruments. The dotted line for a 30% emulsion indicates viscosity measurement from the Brookfield U.L. adapter. The other lines shown on the figure are drawn through the Weissenberg Rheogoniometer data. Up to 70% concentration, there is a good agreement between the different instruments. For oil concentrations greater than 70% (i.e. for 76%, 80% and 82% emulsions), significant deviation is seen between the Weissenberg Rheogoniometer and the Fann viscometer data. The Rheogoniometer gives lower viscosities than the Fann viscometer for the same concentration emulsion at the same shear rate. Figure 2 shows the rheograms (τ vs. $\dot{\gamma}$) of water-in-oil emulsions obtained from different instruments. The dotted lines for 0%, 10% and 20% emulsions indicate viscosity measurements from the Brookfield U.L. adapter. The other lines shown on the figure are drawn through the Weissenberg Rheogoniometer data. Up to 60% concentration, there is an excellent agreement between the different instruments. However, for water concentrations greater than 60%, the flow curves obtained from the different instruments are appreciably different for the same concentration emulsions. Like for oil-in-water emulsions, the Rheogoniometer gives lower viscosities than the Fann viscometer at the same shear rate. Figures 3 and 4 show the flow curves of oil-in-water and water-in-oil emulsions obtained from the Brookfield viscometer. It is quite obvious from the figures that the concentrated emulsions (both type) exhibit some yield stress. Yield stress is also observed in the flow curves obtained from the Fann viscometer. However, no yield stress is observed in the flow curves obtained from the Weissenberg Rheogoniometer.

The shift in the flow curves obtained from the different instruments for the same emulsion and the absence of yield stress in the Rheogoniometer can be explained by "slip effects" in the Rheogoniometer. "Slip effects" are known to exist in capillary tubes and small diameter pipes (3).

When a dispersed system enters the narrow capillary or tube, the dispersed phase shows a tendency to migrate away from the tube wall towards the tube axis. If this occurs there is a concentration fluctuation across the capillary with the major reduction in concentration in the layers adjacent to the capillary wall. This phenomenon has actually been observed with a suspension of paper pulp (4). When this suspension flows through a transparent pipe a clear water annulus is observed adjacent to the wall. The fluid in the region close to the wall has a lower viscosity and this gives rise to an effective velocity of slip at the wall. Different diameter capillaries will then give different flow curves. Oldroyd (5) has done detailed analysis of slip effects in pipes.

It is highly likely that the slip effects would also exist in a cone-and-plate type viscometer. Due to preferred orientation of oil particles, a region adjacent to cone and plate may form which is deficient in dispersed material. This will lead to effective slip at the cone and plate surface resulting in lower viscosities and absence of yield stress. Boardman and Whitmore (6) have also observed similar effects for flocculated china clay suspensions. These suspensions are normally Bingham plastic in nature. However, when these suspensions are studied in a cone and plate viscometer, they appear to behave as pseudo plastic fluids (with no yield stress) and the linear portion of the flow curve (shear stress vs. shear rate) is well below the true flow curve. The authors have attributed this abnormal behaviour to the presence of effective slip at the cone and plate surface.

ACKNOWLEDGEMENTS

Appreciation is expressed to CANMET for a research contract to enable this investigation to proceed.

NOMENCLATURE

R	radius of cone
R_1	radius of inner cylinder (bob) of coaxial cylinder viscometer
R_2	radius of outer cylinder (rotor) of coaxial cylinder viscometer
t	$2\pi/\Omega$, time required for one revolution
T	torque

Greek Letters

α	cone angle
B	R_2/R_1 , ratio of radius of outer cylinder to inner cylinder of coaxial cylinder viscometer
ΔT	transducer reading
ϕ	volume concentration of dispersed phase
γ	shear rate
Ω	angular velocity
τ	shear stress

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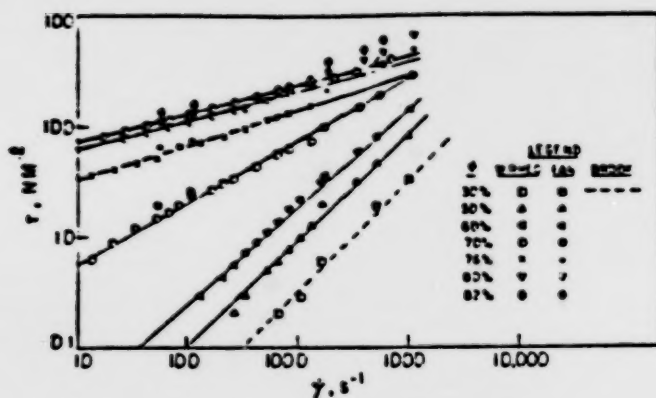


Fig. 1: Rheograms of oil-in-water emulsions

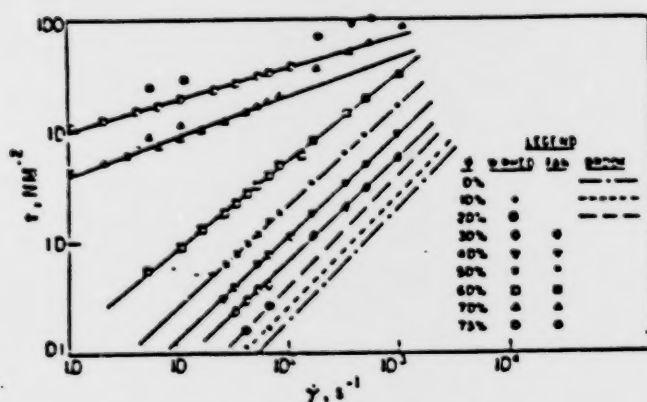


Fig. 2: Rheograms of water-in-oil emulsions

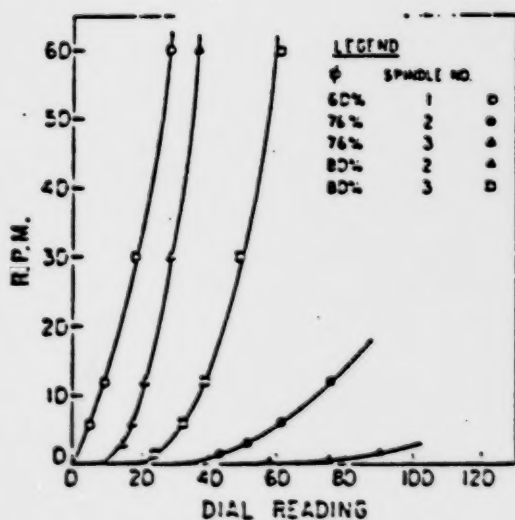


Fig. 3: Brookfield viscometer data for oil-in-water emulsions

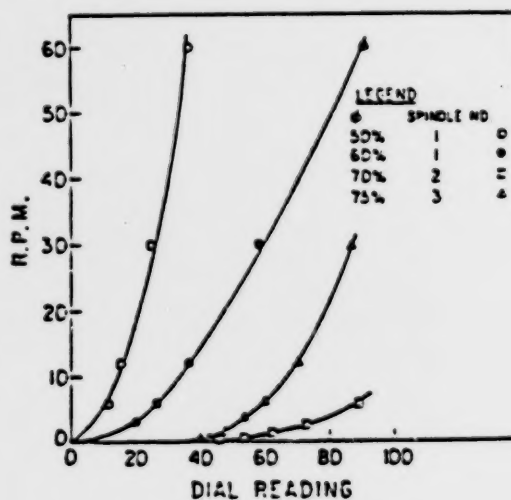


Fig. 4: Brookfield viscometer data for water-in-oil emulsions

PAPER 4

FLOW BEHAVIOUR OF OIL-IN-WATER EMULSIONS

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ABSTRACT

The rheological behaviour of concentrated oil-in-water emulsions has been investigated using the Weissenberg Rheogoniometer. The effects of shear rate and oil concentration on the viscosity have been determined. From the rheological data, the maximum attainable oil concentration in the emulsion has been estimated and is introduced as a parameter in the relationship between relative viscosity and oil concentration to account for certain variations in the emulsion characteristics. Empirical equations have been obtained which correlate zero shear viscosity and infinite shear viscosity as a function of reduced oil concentration. Estimated maximum attainable oil concentration is found to be in close agreement with the actual maximum concentration that could be achieved experimentally.

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INTRODUCTION

Crude oils produced from secondary and tertiary oil recovery projects are very often in the form of stable oil-in-water emulsions. Oil-in-water emulsions are also encountered in primary production but this is relatively rare. Very often these emulsions are deliberately produced to reduce the viscosity of highly viscous crude oils so that they can be transported easily through the pipelines. Two commercial emulsion pipeline systems for crude transport are known. One in Indonesia (Sommer and Simpson, 1963), carries 6359.6 m³/day of a 70 percent (volume) emulsion of a high pour point waxy crude in a 0.508 m diameter pipeline which is 238 km long. The other, in California (Marsden and Raghavan, 1973), carries a 50 percent emulsion of highly viscous 13.5 API gravity crude 20.92 km in a 0.2032 m diameter pipeline. Understanding the rheological behaviour of these emulsions is of interest for the design of pipelines, mixing equipment and heating equipment, etc.

The broad objective of this study was to investigate the flow behaviour of concentrated emulsions. Specific objectives included:

1. Determination of a relationship between relative viscosity and oil concentration.
2. Estimation of maximum attainable dispersed phase concentration.
3. Determination of relationships between relative viscosity and reduced oil concentration.
4. Comparison of zero shear viscosity data with two phase solid-liquid suspensions.
5. Study of emulsion flow characteristics near the maximum attainable concentration.

PREVIOUS WORK

Mao and Marsden (1977) studied the stability of concentrated crude

oil-in-water emulsion as a function of shear rate, temperature and oil concentration. Their purpose was to determine the rheological properties of emulsions containing different amounts of crude oil at different temperatures. In addition, they also studied the conditions of temperature, composition and shear under which emulsion inversion occurred. From a theoretical consideration, Richardson (1933) first obtained an expression for viscosity of emulsion as follows:

$$\mu_r = \exp (K_1 \phi) \quad (1)$$

Simon and Poynter (1968) used Richardson's expression to correlate the flow behaviour of crude oil-water emulsions stabilized by a non-ionic surfactant. They obtained a value for K_1 equal to 7.0 for ϕ less than 0.74 and 8.0 for greater than 0.74. However, this expression does not take into account the particle size effects and non-Newtonian nature of concentrated emulsions.

Sherman (1960a, 1965b) noted a large effect of particle size on the viscosity of oil-in-water emulsions. He obtained relationship between relative viscosity and interparticle distance. Oldroyd (1953a, 1955b) and Rajagopal (1960) have also proposed viscosity correlations for dilute emulsions incorporating the particle size effect. However, there has been no attempt to relate the non-Newtonian viscosity of the emulsion to the dispersed phase concentration except using the limiting viscosity and develop a unified relationship for various particle sizes.

EXPERIMENTAL

The oil used in the emulsion preparation was a white mineral oil supplied by Imperial Oil Limited. Tap water was used in all emulsion preparations.

Non-ionic emulsifier Triton x-100 (iso-octyl phenoxy polyethoxy ethanol) supplied by Rohm and Haas Co. (USA) was used to stabilize the emulsions. For Set A, 0.2% by volume of emulsifier based on water present in the emulsion was used and for Set B, 1% by volume of emulsifier was used. The method of preparation was the agent-in-water method where the emulsifier was first dissolved in water and continuously agitated while the oil was slowly added and dispersed in the body of the emulsion by the agitator. The emulsion was prepared in a baffled stainless steel vessel where a disc type stirrer at a controlled speed was used. Photomicrographs of different concentration emulsions were taken using a Zeiss microscope, in which transmitted light was used. A small sample of emulsion was diluted 20 to 30 times by glycerol and the diluted sample was placed on a dimple slide for study in the microscope.

A Weissenberg Rheogoniometer with cone and plate geometry was used to measure the shear stress-shear rate relationship. The platen diameter was 100 mm and cone angle was 4° , giving a clearance between the cone flat (truncated tip) and adjacent plate of $171 \mu\text{m}$. The flat platen of the rheogoniometer was replaced by a newly made platen with dished end to facilitate the use of emulsions in the instrument. This helped to retain the test material in the cone and plate assembly and data at higher shear rate could be obtained without any loss of material which could otherwise have occurred. The dished platen was calibrated and the experimental data were corrected for edge effects. The temperature of the emulsion was kept constant at $21 \pm 0.5^\circ\text{C}$.

RESULTS AND DISCUSSIONS

The shear stress-shear rate relationships for mineral oil-in-water emulsions has been studied with a maximum oil concentration of 94 percent by volume at a constant temperature of 21°C . Data on two sets of emulsions are presented where respectively 0.2 and 1 percent by volume of emulsifier based on

water were used. The maximum concentrations used in the experiments were realistically the highest concentration that could be obtained without instability occurring in the emulsion. The shear rate range covered in the experiments were from 0.0271 to 538 sec^{-1} . For low concentration emulsions, measurement inaccuracies were observed at very low and very high shear rates. On the other hand, for concentrated emulsions, exudation of materials from cone and dish assembly occurred only at very higher shear rates. To study the effect of concentration on flow behaviour oil concentrations ranging from 40% to 84% were used.

Flow Behaviour Model

All the experimental shear stress-shear rate data were fitted to the Power law model for shear rates greater than 2 sec^{-1} . For the power law equation given below

$$\tau = K \dot{\gamma}^n \quad (2)$$

the parameters k and n were estimated for all the emulsions and are tabulated in Table 1. The rheograms for one set of concentrations (Set B) are given in Figure 1. The rheograms were found to follow linear relationships if only the data above 200 sec^{-1} were considered. A Bingham type model could be fitted to those data as follows:

$$\tau = \tau_y + \eta \dot{\gamma} \quad (3)$$

τ_y and η values calculated for the rheograms are given in Table 2. These values have been used for some correlations as will be shown in a later section.

Apparent Viscosities for Emulsions

As most of the emulsions exhibited non-Newtonian behaviour apparent viscosity has to be determined for use in the correlations. Since the apparent viscosity is a function of shear rate it has to be estimated at various shear rates. In this study it was decided to estimate the apparent viscosity at 0, 100 and 400 sec^{-1} (covering a wide range of shear rates up to a high value). In addition η was used to correlate the data at high shear rates in place of the apparent viscosity at high shear rate (μ_a). A plot of μ_a versus $\dot{\gamma}$ is given in Figure 2 from which zero shear rate viscosity was obtained by extrapolating μ_a to zero shear rate.

Correlation of μ_a and ϕ

For solid-liquid suspensions a number of empirical and semi-empirical relationships are available relating viscosity of suspensions to the volume concentration of solids. Chong et al. (1971), studied the concentration effect of neutrally buoyant spherical particles on suspension viscosity and proposed the following correlation to fit their data on glass bead-polyisobutylene suspension.

$$\mu_y = (1 + A \frac{\phi/\phi_m}{1 - \phi/\phi_m})^B \quad (4)$$

where $A = 0.75$ and $B = 2$ are two parameters.

Relationships for μ_y and ϕ_n in the high ϕ -range were obtained by Frankel and Acrivos (1967) and Eiler (1941) also. But all these expressions were valid only for Newtonian suspensions. In case of non-Newtonian suspensions relative viscosities would have to be estimated on the basis of apparent viscosities. Ghosh and Bhattacharya (1982) in a recent study of coal-oil

suspension rheology used zero shear viscosity to calculate relative viscosity. In this study relative viscosity has been estimated at 0, 100 and 400 sec^{-1} shear rates. In addition η has also been used to determine relative viscosity at high shear rate.

A plot of ν_γ versus ϕ at different shear rates is shown in Figure 3. The relationship shown in Figure 3 is similar to the solid-liquid suspension behaviour in that at higher concentration, the viscosity increases dramatically and becomes almost asymptotic at a very high concentration. However, these relations are very much affected by the particle size and size distribution of oil particles in the emulsion which means for different batches of emulsions ν_γ will be different at the same shear rate and ϕ . (In another paper, See #3, "Slip Effects", it has been shown that different viscometers can give different results due to slip effects at the walls of the instrument).

Particle Size and Size Distribution

Figure 4 (for Set A) and Figure 5 (for Set B) show the typical photomicrographs of various concentrated emulsions. It can be seen that the droplet size and size distribution does not vary significantly with the dispersed phase concentration. Variation in particle size is, however, seen for the same concentration emulsions having different emulsifier content.

Maximum Volume Concentration

Maximum volume concentration attainable in a dispersion may be influenced by many factors such as particle size, size distribution, shape, particulate surface charge, interaction between particles and medium, concentration of emulsifier, etc. For solid-liquid suspensions, many workers have used ϕ_m to take into account the variation of particle size and size distribution and obtained correlations in terms of ν_γ and ϕ/ϕ_m . Recently Low and Bhattacharya (1983) have demonstrated that with the introduction of ϕ_m , ν_γ

data for coal-oil suspensions of different moisture concentrations could be expressed by a single curve of ν_Y versus ϕ/ϕ_m . In this study also, ϕ_m has been used to correlate all viscosity data. ϕ_m was determined from the experimental data of ν_Y and ϕ using Thomas's method (1965) of plotting $(\nu_Y - 1)^{-1}$ versus γ and extrapolating the curve to a zero value of $(\nu_Y - 1)^{-1}$. In Figure 6 a plot of $(\nu_Y - 1)^{-1}$ versus ϕ is shown where ν_Y has been given for different shear rates. It is interesting to see that extrapolations for various shear rates produce the same value for ϕ_m . In this case ϕ_m is estimated to be 0.905 for Set A and 0.89 for Set B. Experimentally it was observed that stable emulsion of ϕ higher than 0.9 could not be prepared for Set A and for Set B the limit was around 0.88. Beyond this concentration it was noted that a separate oil phase appeared in conjunction with the oil-in-water emulsion phase. It is presumed that emulsion becomes saturated and rather unstable near its maximum concentration where coalescence can easily work and therefore where this limit is exceeded there exist two phases namely oil and oil-in-water emulsion. This point of maximum concentration and instability may, therefore, be estimated from the method described.

ν_Y versus ϕ/ϕ_m

In Figure 7 relative apparent viscosity is plotted against ϕ/ϕ_m as a function of shear rate. Data are presented for the shear rates of 0, 100 and 400 sec^{-1} respectively and high shear rate data have also been expressed in terms of η/η_c where η is the plastic viscosity given in Table 2. It is obvious from Figure 7 that ν_Y versus ϕ/ϕ_m produces a series of curves within the limits of zero shear rate data and the plastic viscosity data.

The relative viscosity at zero shear rate is about two orders of magnitude higher than those corresponding to 100 sec^{-1} . This is because of the existence of large number of flocs of oil globules at zero shear rate. In

Figure 8 μ_Y at zero shear rate has been compared with the empirical expression of Ghosh and Bhattacharya (1982) for black coal-oil suspension as given below:

$$\mu_Y = (1 + 0.163 \frac{\phi/\phi_m}{1 - \phi/\phi_m})^{9.3} \quad (5)$$

Comparison has also been made with the empirical expression for Chong et al. (1971), given in Equation (4). It can be seen in Figure 8 that μ_Y values for emulsions are much higher than those for the coal-oil suspensions for the same values of ϕ/ϕ_m . This may be due to the fact that flocculation is quite significant in case of emulsions, particularly at high dispersed phase concentrations, resulting in higher viscosities. The other contributing factor could be that although oil globules in the emulsion might behave as rigid particles the interfacial viscosity, because of the presence of the emulsifier, will be much higher than the continuous phase viscosity thus giving rise to higher viscosity in case of emulsions. Coal-oil suspensions have been observed to agglomerate heavily only at very high concentration. The data of Chong et al. (1971), as shown in Figure 8, are much lower than the present work particularly at high concentrations. This is because Chong's expression is valid only for Newtonian non-interacting systems. Except for the data of Ghosh and Bhattacharya, no other data in literature was found to compare with the present emulsion data. The following empirical relationships were obtained relating zero shear rate viscosity and ϕ/ϕ_m :

$$\mu_Y = 1 + 7.92 \left(\frac{\phi/\phi_m}{1 - \phi/\phi_m} \right)^{1.21} \quad (6)$$

for $0 \leq \phi/\phi_m \leq 0.66$

and

$$\nu_Y = 2.4 \times 10^{-8} \exp (13.75 \phi/\phi_m) \quad (7)$$

for $\phi/\phi_m > 0.66$

Correlation for High Shear Viscosity

The high shear data have been expressed by means of the Bingham Plastic relation as shown in Equation (3). The plastic viscosity η , which is independent of shear rate can be used to estimate relative viscosity in place of apparent viscosity. The relative plastic viscosity η_Y , has been plotted against ϕ/ϕ_m in Figure 9 where the data from both Set A and Set B have been included. It is interesting to note that data from both the sets fall on the same curve. Regression analysis gave the following best fit relationship for the data:

$$\eta_Y = 0.84 \exp (2.07 \phi/\phi_m) \quad (8)$$

However since equation (8) does not reduce to 1 when ϕ/ϕ_m equals zero, an alternative relationship was also obtained as follows:

$$\eta_Y = \exp (1.95 \phi/\phi_m) \quad (9)$$

where η_Y becomes equal to 1 for ϕ/ϕ_m equal to zero. Equation (8) is found to fit the data more accurately, particularly for ϕ/ϕ_m greater than 0.6. There is no data available in literature to compare with these results. Simon and Poynter (1968) obtained a similar expression with a much different co-efficient. However, they used the viscosity at infinite shear rate which cannot be considered constant. Thus, it was not possible to compare their results with the present correlation.

Emulsion Stability

Emulsions with concentration above 90 percent were prepared to investigate the stability of the emulsion at very high concentration. Rheogram of a 90.4 percent emulsion (having 1% by volume emulsifier based on water phase) is given in Figure 10. When the oil concentration in the emulsion was raised above 90.4 percent, no further rise in viscosity was observed and in the rheogoniometer a separation was found to occur between oil and emulsion phase at shear rates above only 6 sec^{-1} . When the oil concentration was increased to 95 percent, a clear separation of oil and emulsion phase was visible in the emulsifying vessel itself. These two phases were separated from the above mixture and the rheograms for the two phases are given in Figure 10. The viscosity of the emulsion phase is lower than that for 90.4 percent stable emulsion indicating that the emulsion phase is unsaturated. The separated oil phase exhibits a viscosity greater than that for pure oil as shown in Figure 10. It was observed that this oil phase contained small concentrations of oil-in-water emulsion and water as dispersed particles.

CONCLUSIONS

Based on the experimental observations and the analysis of the data, the following conclusions are reached:

1. The mineral oil-in-water emulsions behave as time independent non-Newtonian fluids at higher dispersed phase concentrations.
2. Emulsion viscosity increases sharply with the increase in volume concentration of dispersed phase.
3. Comparison of zero shear viscosity of emulsion with that of solid-liquid suspension indicates that oil-in-water emulsions exhibit higher viscosity until a very high concentration of the dispersed phase is reached where both become closer.

4. The relationship between the relative viscosity and the dispersed phase concentration will vary with the variation in emulsion characteristics such as particle size, emulsifier concentration, etc. The variation in emulsion characteristics can be taken into account by introduction of ϕ_m , the maximum attainable dispersed phase concentration. A unique correlation is, therefore, obtained between the relative viscosity and reduced dispersed phase concentration.
5. Based on experimental data, empirical equations which correlate zero shear viscosity and infinite shear viscosity as a function of reduced oil concentration, are proposed.
6. Emulsion appears to be unstable near its maximum attainable concentration. Estimated maximum concentration is very close to actual maximum concentration that could be achieved experimentally.

ACKNOWLEDGEMENT

Appreciation is expressed to CANMET for a research contract to enable this investigation to proceed.

Nomenclature

A	constant in Equation 4, dimensionless
B	constant in Equation 4, dimensionless
K	power law constant, $\text{NM}^{-2} \text{s}^n$
K_1	constant in Equation 1, dimensionless
n	flow behaviour index, dimensionless
$\frac{du}{dr}$	shear rate, s^{-1}

Greek Letters

η	plastic viscosity, $\text{NM}^{-2} \text{s}$
η_r	relative plastic viscosity η/η_c , dimensionless
ϕ	dispersed phase concentration, dimensionless
ϕ_m	maximum attainable dispersed phase concentration, dimensionless
$\dot{\gamma}$	shear rate, s^{-1}
η_a	apparent viscosity, $\text{NM}^{-2} \text{s}$
$\eta_{a\infty}$	apparent viscosity at high shear rate, $\text{NM}^{-2} \text{s}$
η_c	viscosity of continuous phase, $\text{NM}^{-2} \text{s}$
η_r	relative viscosity η/η_c , dimensionless
τ	shear stress, NM^{-2}
τ_y	yield stress, NM^{-2}

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Table 1
Power Law Constants

SET - A (0.2% by vol. emulsifier)			SET - B (1% by vol. emulsifier)		
ϕ	K ($\text{NM}^{-2} \text{s}^n$)	n	ϕ	K ($\text{NM}^{-2} \text{s}^n$)	n
0.45	0.0078	1.01	0.4	0.007	1.01
0.55	0.017	0.96	0.5	0.014	0.958
0.65	0.235	0.73	0.6	0.06	0.829
0.68	1.515	0.49	0.65	0.278	0.672
0.71	1.655	0.49	0.72	2.053	0.448
0.76	4.67	0.35	0.80	12.54	0.27
0.80	5.93	0.34	0.84	22.95	0.263

Table 2
Bingham Model Constants

SET - A (0.2% by vol. emulsifier)			SET - B (1% by vol. emulsifier)		
ϕ	τ_y (NM ⁻²)	n (NM ⁻² s)	ϕ	τ_y (NM ⁻²)	n (NM ⁻² s)
0.65	6.7	0.0277	0.60	0.5	0.0209
0.68	13.75	0.0362	0.65	3.5	0.03
0.71	14.25	0.040	0.68	9.5	0.0344
0.76	20.75	0.048	0.72	15.0	0.0362
0.80	32.75	0.049	0.76	28.5	0.0373
			0.80	40.5	0.0616
			0.84	74	0.0836

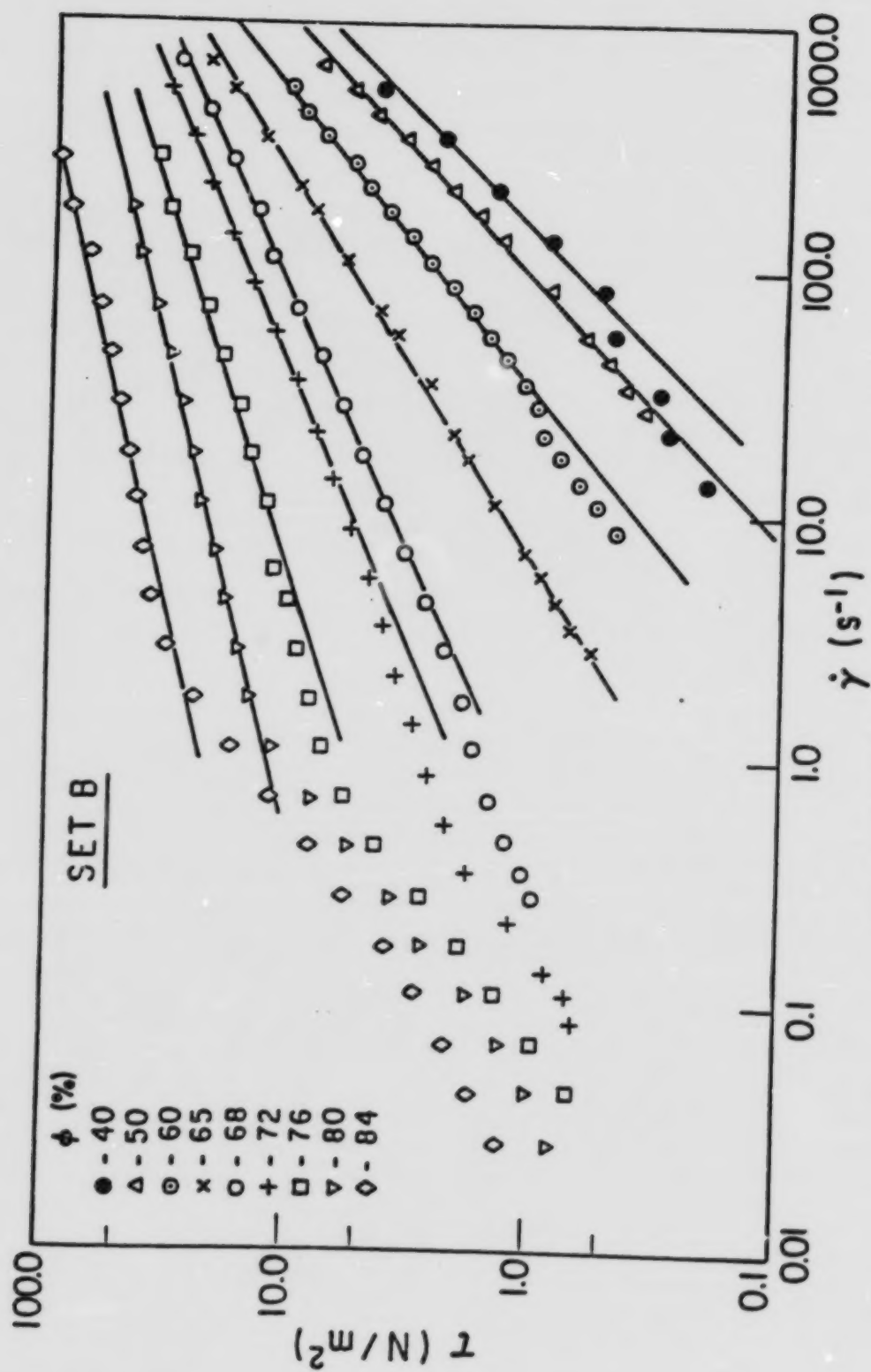


Figure 1: RHEOGRAMS OF OIL-IN-WATER EMULSIONS

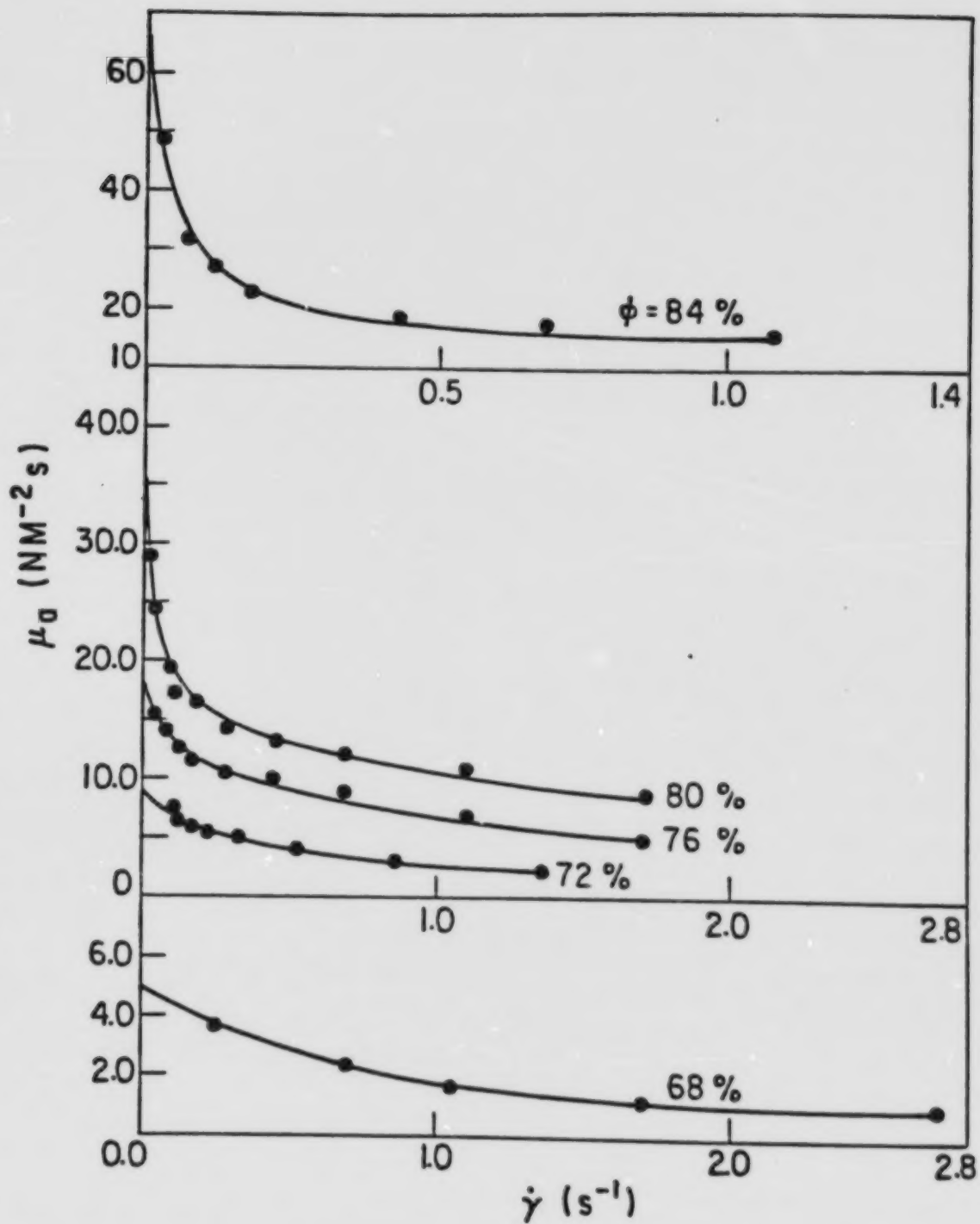


Figure 2: ESTIMATION OF ZERO SHEAR VISCOSITY

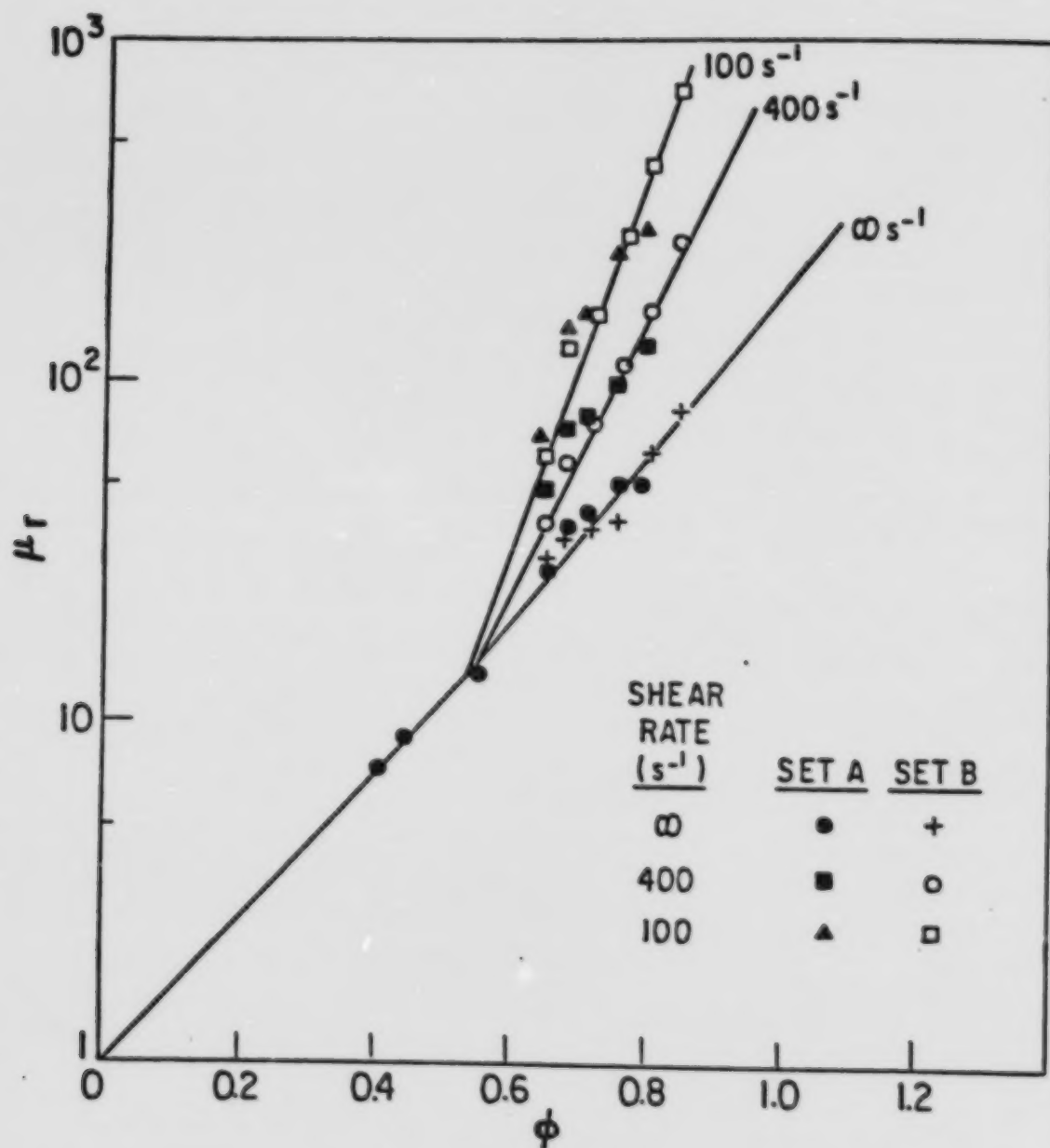
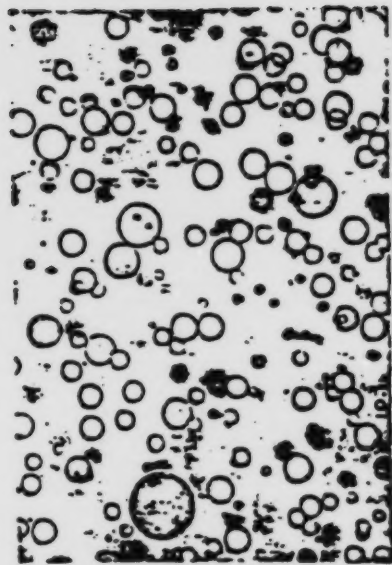


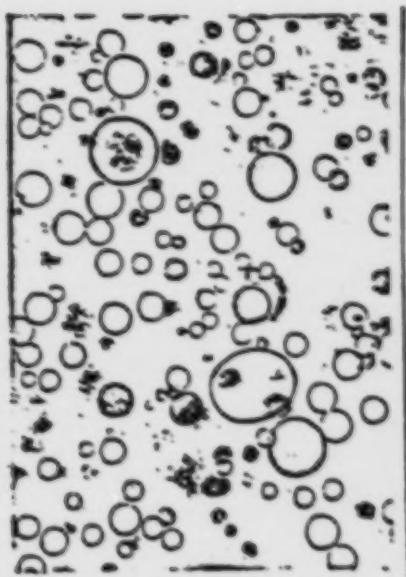
Figure 3: RELATIVE VISCOSITY VS OIL CONCENTRATION



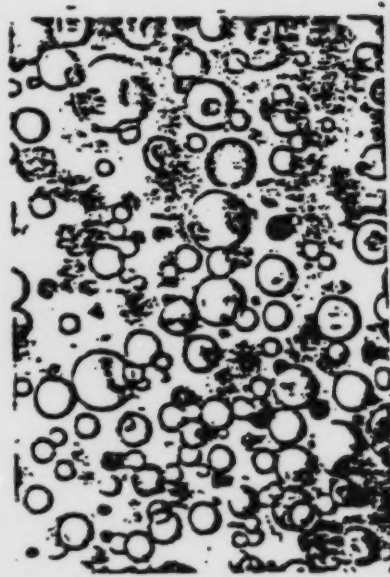
Set A: 55% OIL-IN-WATER
EMULSION



Set A: 65% OIL-IN-WATER
EMULSION



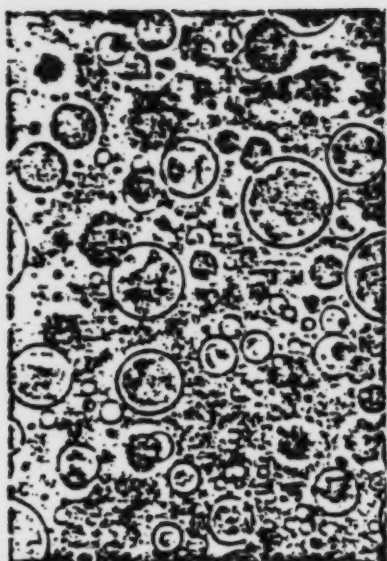
Set A: 65% OIL-IN-WATER
EMULSION



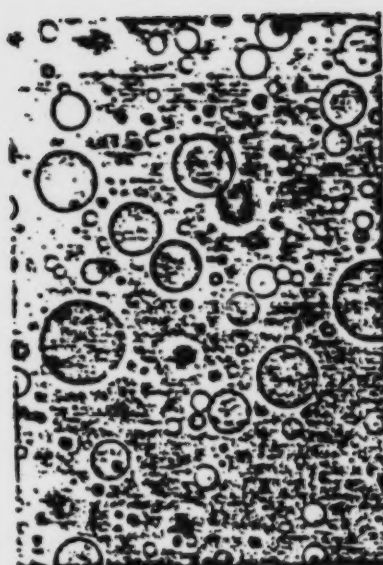
Set A: 80% OIL-IN-WATER
EMULSION

Figure 4: Photomicrographs of Set A

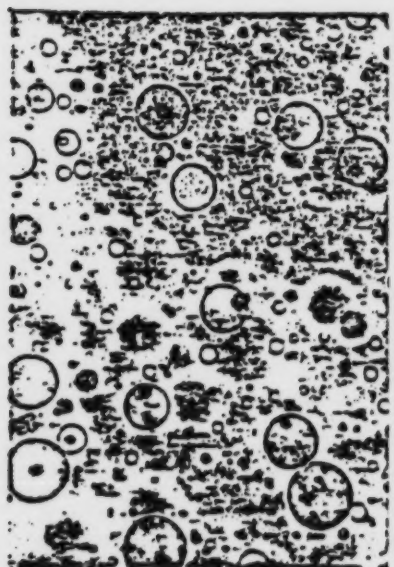
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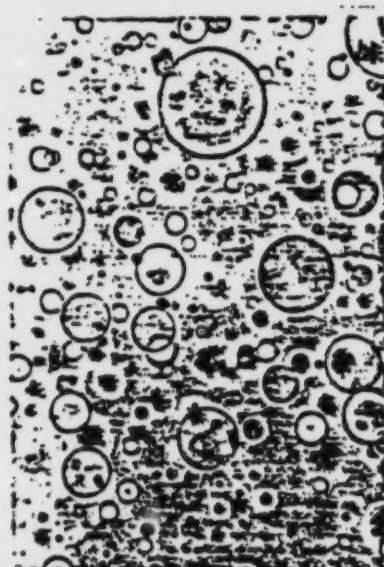
Set B: 60% OIL-IN-WATER
EMULSION



Set B: 68% OIL-IN-WATER
EMULSION



Set B: 65% OIL-IN-WATER
EMULSION



Set B: 72% OIL-IN-WATER
EMULSION

Figure 5: Photomicrographs of Set B

5

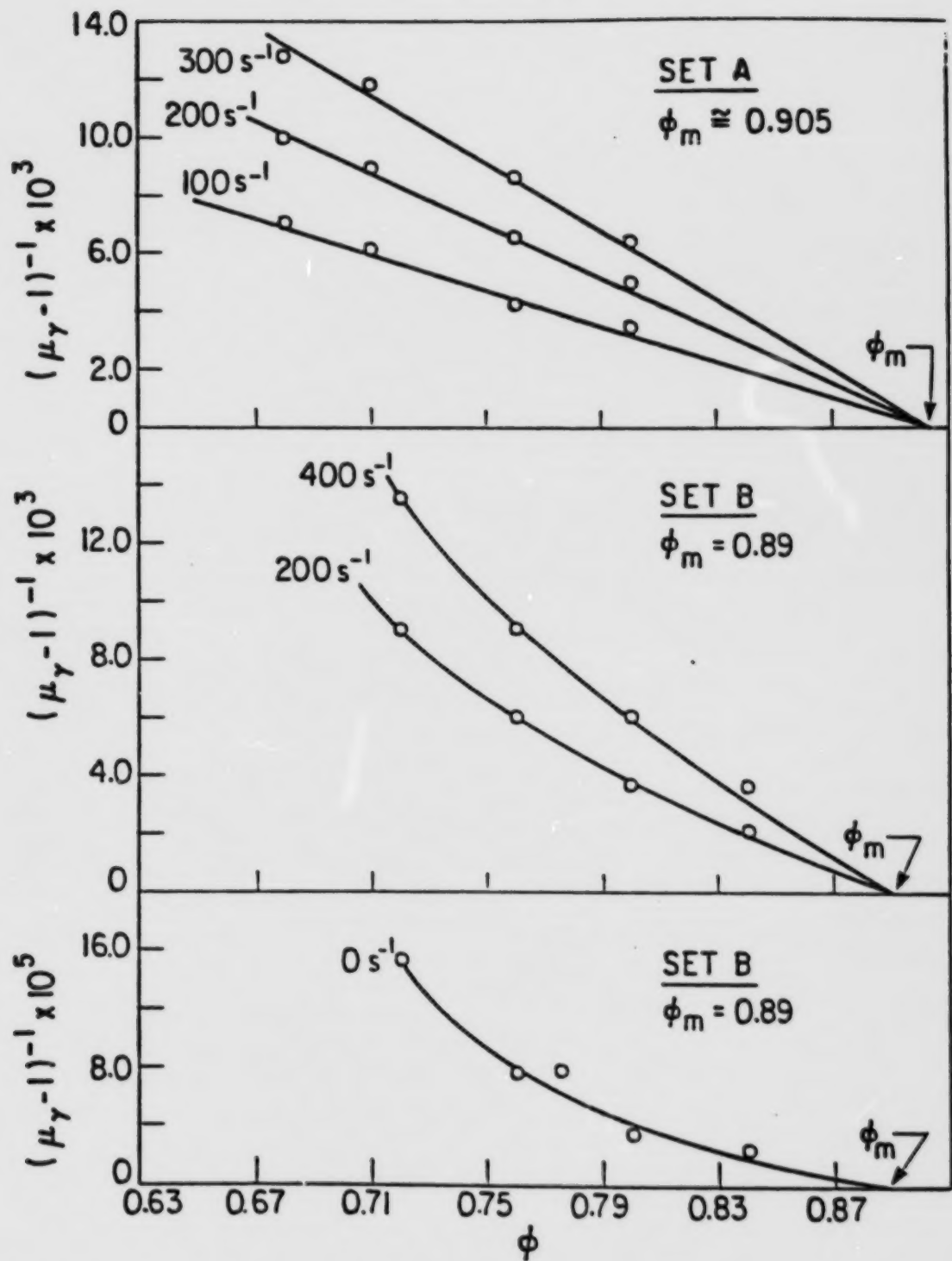
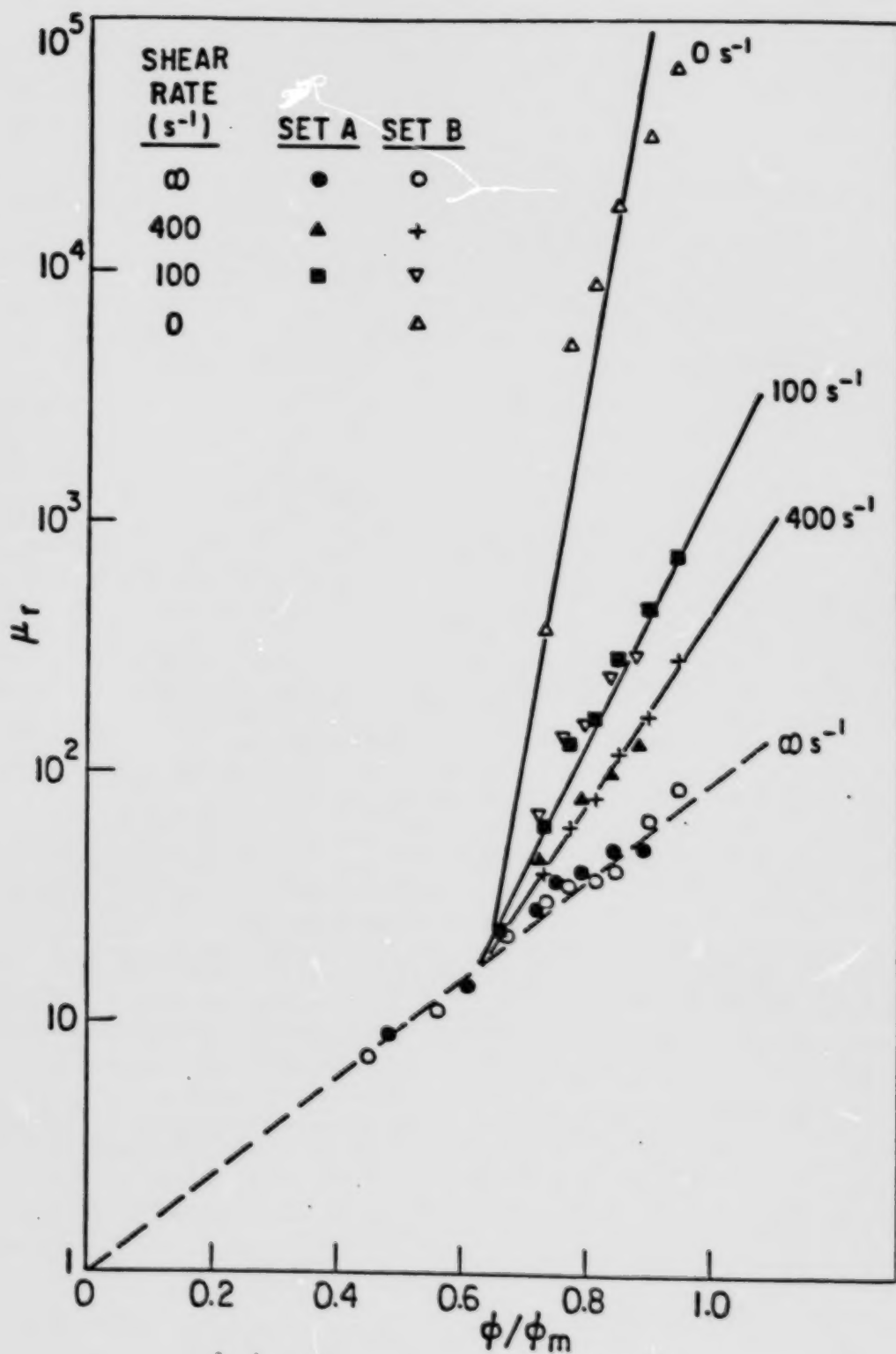


Figure 6 ESTIMATION OF MAXIMUM
 ATTAINABLE OIL CONCENTRATION



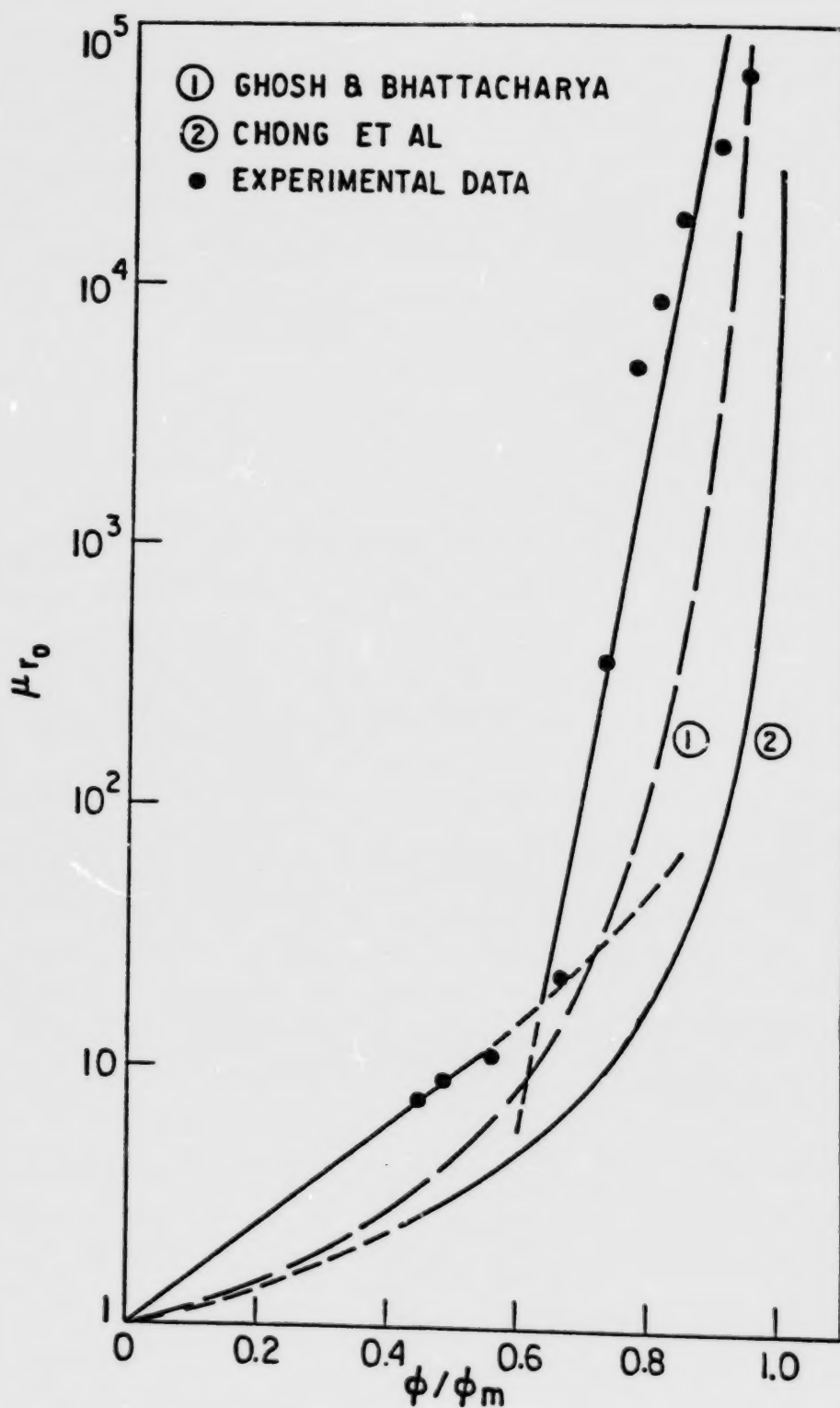


Figure 8. COMPARISON OF ZERO SHEAR VISCOSITY OF EMULSIONS WITH SOLID-LIQUID SUSPENSIONS

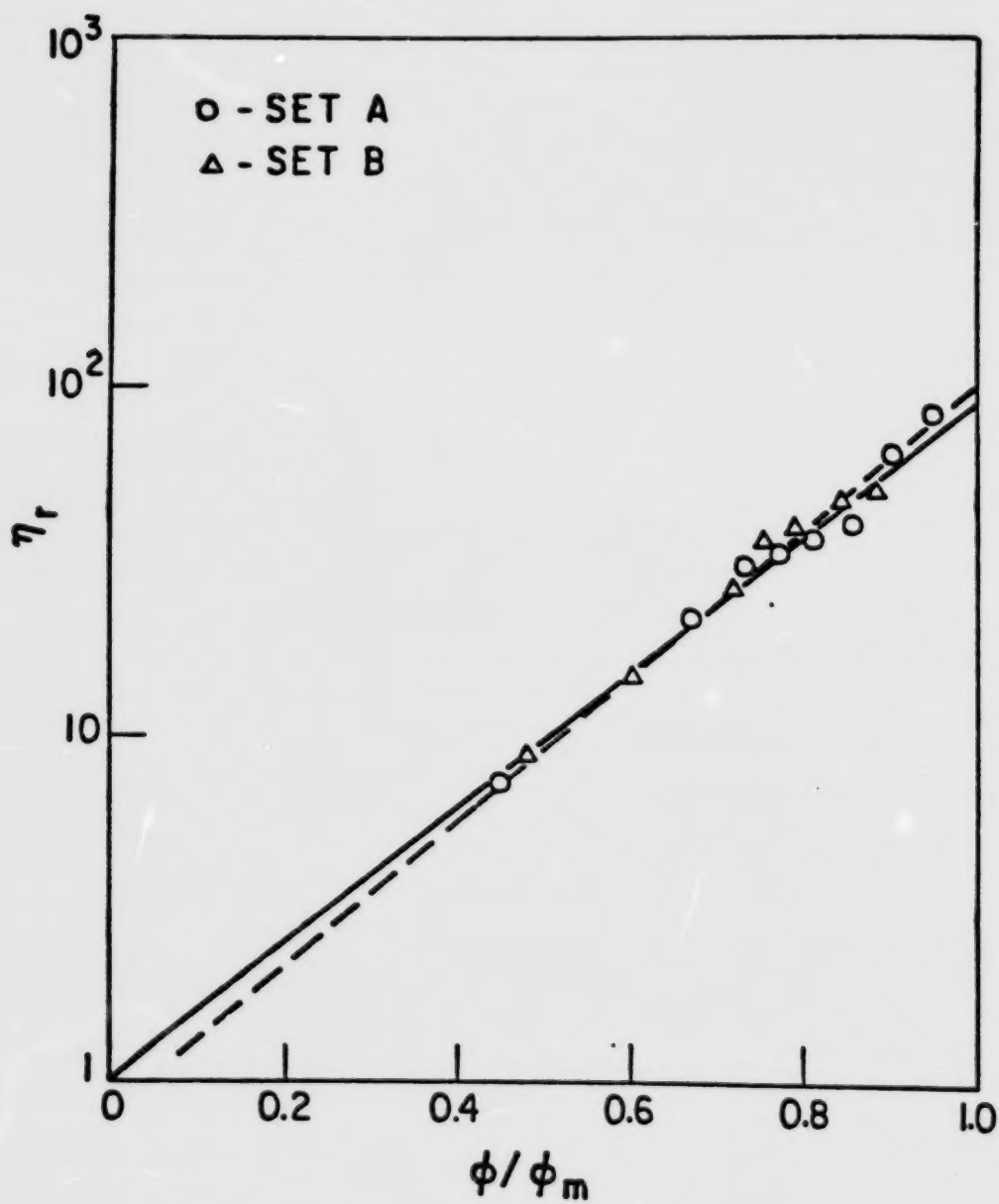


Figure 9: RELATIVE PLASTIC VISCOSITY
VS REDUCED OIL CONCENTRATION

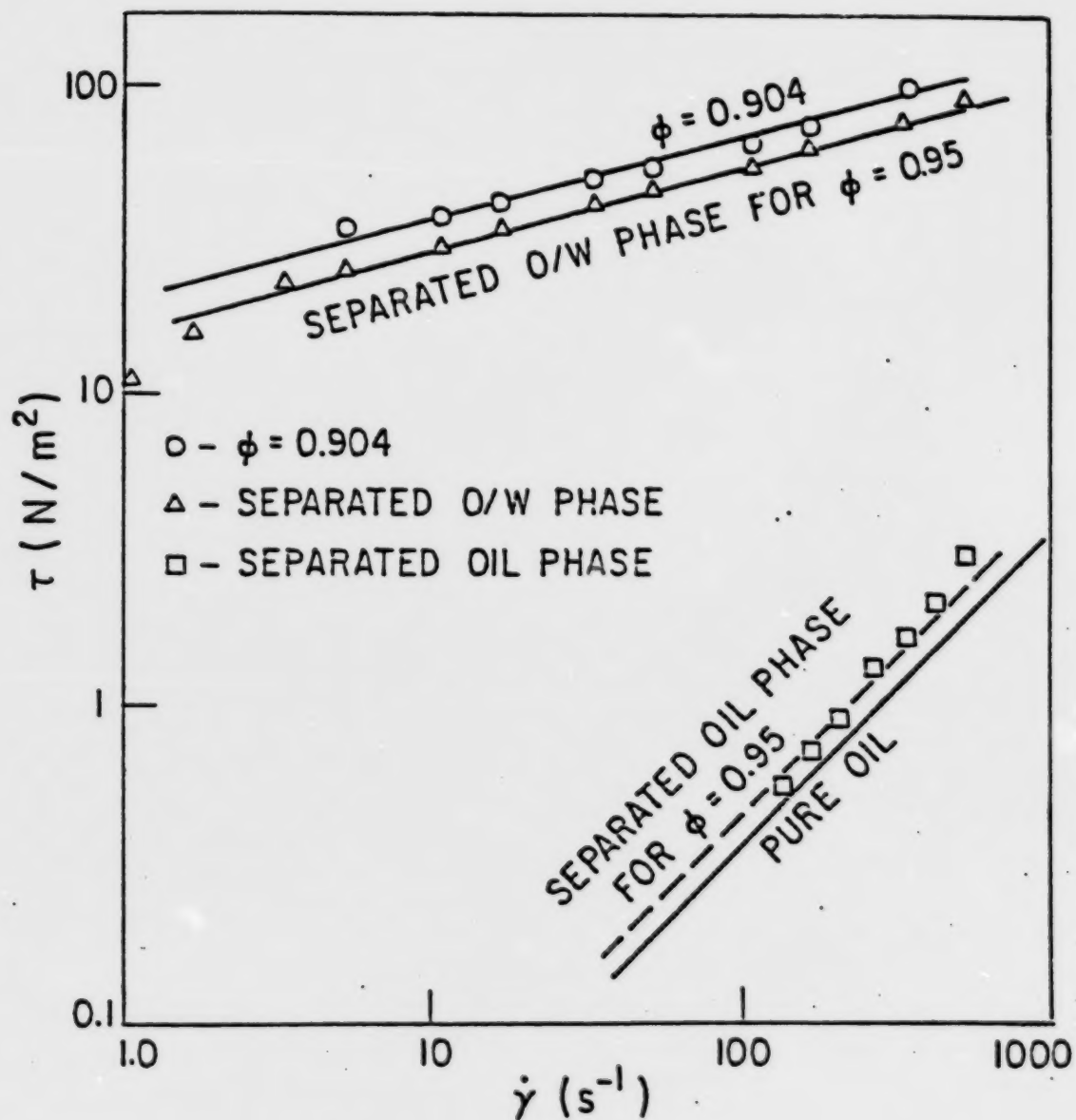


Figure 10: Rheograms of emulsion before and after phase separation

PAPER 5

RHEOLOGICAL STUDY OF CONCENTRATED EMULSIONS

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ABSTRACT

The flow behaviour of concentrated mineral oil-in-water emulsions has been investigated using the Weissenberg Rheogoniometer at ambient temperature. Emulsions have been prepared with the help of a non-ionic emulsifying agent Triton x-100. At higher concentrations these emulsions have been found to be highly non-Newtonian in nature, although no measurable normal stress has been observed for any of these emulsions.

Relationships have been obtained between relative apparent viscosity and oil concentration at various shear rates. The maximum attainable concentration of oil in the emulsion has been estimated from the rheological data and is introduced as a parameter in the relationship between relative viscosity and oil concentration to account for certain variations in the emulsion characteristics. Results for zero shear viscosity have been compared with those available for solid-liquid suspensions. Empirical relationships have been obtained for relative viscosity at high shear rates.

KEYWORDS

Rheology; emulsion; emulsion rheology; oil-in-water emulsion; flow behaviour.

INTRODUCTION

Many crude oils are produced as emulsions and there are many products in industries which are also produced as oil-in-water emulsions. The stability and flow behaviour of oil-water emulsions are considerably influenced by the concentration of oil and the nature of emulsifying agents present in the emulsion. Rheological study of these emulsions is therefore very important for the design of systems involving transportation, storage, mixing, pumping, cooling, heating, etc.

The objective of this work was to investigate the flow behaviour of oil-water emulsions at higher concentrations, determine a relationship between relative viscosity and oil concentration and compare it with two-phase solid liquid suspension behaviour. A further objective was to study the emulsion flow characteristics near its maximum concentration and estimate this maximum concentration from the rheological data.

EXPERIMENTAL

The oil used in the emulsion preparation was a white mineral oil supplied by Imperial Oil Limited. Tap water was used in all emulsion preparations. Non-ionic emulsifier Triton x-100 (iso-octyl phenoxy polyethoxy ethanol) supplied by Rohm and Haas Co. (USA) was used to stabilize the emulsions. For Set A, 0.2% by volume of emulsifier based on water present in the emulsion was used and for Set B, 1% by volume of emulsifier was used. The method of preparation was the agent-in-water method where the emulsifier was first dissolved in water and continuously agitated while the oil was slowly added and dispersed in the body of the emulsion by the agitator. The emulsion was prepared in a baffled stainless steel vessel where a disc type stirrer at a controlled speed was used. Photomicrographs of different concentration emulsions were taken using a Zeiss microscope, in which transmitted light was used. Small sample of emulsion was diluted 20 to 30 times by glycerol and the diluted sample was placed in a dimple slide for study in the microscope.

A Weissenberg Rheogoniometer with cone and plate geometry was used to measure the shear stress-shear rate relationships. The platen diameter was 100 mm and cone angle was 4°, giving a clearance between the cone flat (truncated tip) and adjacent plate of 171 μm . The flat platen of the rheogoniometer was replaced by a newly made platen with dished end to facilitate the use of emulsions in the instrument. This helped to retain the test material in the cone and plate assembly and data at higher shear rate could be obtained without any loss of material which could otherwise have occurred. The dished platen was calibrated and the experimental data were corrected for edge effects. The temperature of the emulsion was kept constant at $21 \pm 0.5^\circ\text{C}$.

RESULTS AND DISCUSSIONS

The shear stress-shear rate relationships for mineral oil-in-water emulsions have been studied with a maximum oil concentration of 94 percent by volume at a constant temperature of 21°C . Data on two sets of emulsions are presented where respectively 0.2 and 1 percent by volume of emulsifier based on water were used. The maximum concentrations used in the experiments were realistically the highest concentration that could be obtained without instability occurring in the emulsion. The shear rate range covered in the experiments were from 0.0271 to 538 sec^{-1} . For low concentration emulsions, measurement inaccuracies were observed at very low and very high shear rates. On the other hand, for concentrated emulsions, exudation of materials from cone and dish assembly occurred only at very higher shear rates. To study the effect of concentration on flow behaviour oil concentrations ranging from 40% to 84% were used.

Flow Behaviour Model

All the experimental shear stress-shear rate data were fitted to the Power law model for shear rates greater than 2 sec^{-1} . For the power law equation given below

$$\tau = k \left(\frac{du}{dr} \right)^n \quad (1)$$

the parameters k and n were estimated for all the emulsions and are tabulated in Table 1. The rheograms for one set of concentrations (Set B) are given in Fig. 1. The rheograms were found to follow linear relationships if only the data above 200 sec^{-1} were considered. A Bingham type model could be fitted to those data as follows:

$$\tau = \tau_y + n \frac{du}{dr} \quad (2)$$

τ_y and n values calculated for the rheograms are given in Table 2. These values have been used for some correlations as will be shown in a later section.

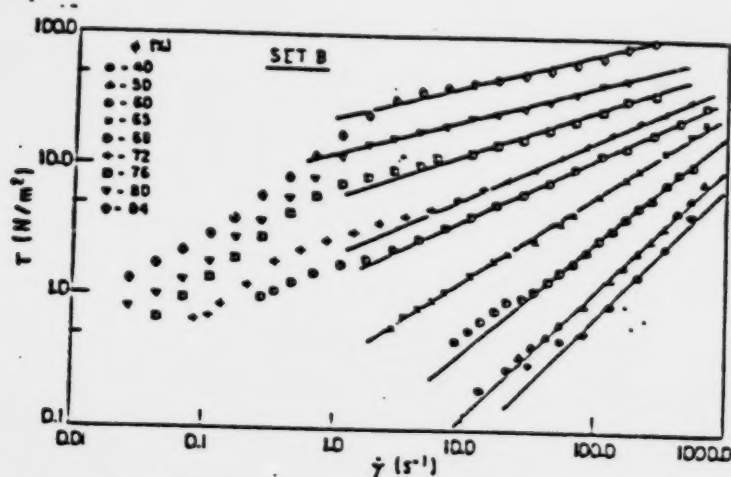


Fig. 1 Rheograms

TABLE 1 Power Law Constants

SET A (0.2% by vol. emulsifier)			SET B (1% by vol. emulsifier)		
ϕ	K	n	ϕ	K	n
0.45	0.0078	1.01	0.4	0.007	1.01
0.55	0.017	0.96	0.5	0.014	0.958
0.65	0.235	0.73	0.6	0.06	0.829
0.68	1.515	0.49	0.65	0.278	0.672
0.71	1.655	0.49	0.72	2.053	0.448
0.76	4.67	0.35	0.80	12.54	0.27
0.80	5.93	0.34	0.84	22.95	0.263

TABLE 2 Bingham Model Constants

SET A (0.2% by vol. emulsifier)			SET B (1% by vol. emulsifier)		
ϕ	τ_y	n	ϕ	τ_y	n
0.65	6.7	0.0277	0.60	0.5	0.0209
0.68	13.75	0.0362	0.65	3.5	0.03
0.71	14.25	0.040	0.68	9.5	0.0344
0.76	20.75	0.048	0.72	15.0	0.0362
0.80	32.75	0.049	0.76	28.5	0.0373
			0.80	40.5	0.0616
			0.84	74	0.0836

APPARENT VISCOSITIES FOR EMULSIONS

As most of the emulsions exhibited non-Newtonian behaviour apparent viscosity has to be determined for use in the correlations. Since the apparent viscosity is a function of shear rate it has to be estimated at various shear rates. In this study it was decided to estimate the apparent viscosity at 0, 100 and 400 sec^{-1} . In addition n was used to correlate the data at high shear rate in place of apparent viscosity at high shear rate (μ_{∞}). A plot of μ_a versus $\dot{\gamma}$ is given in Fig. 2 from which zero shear rate viscosity was obtained by extrapolating μ_a to zero shear rate.

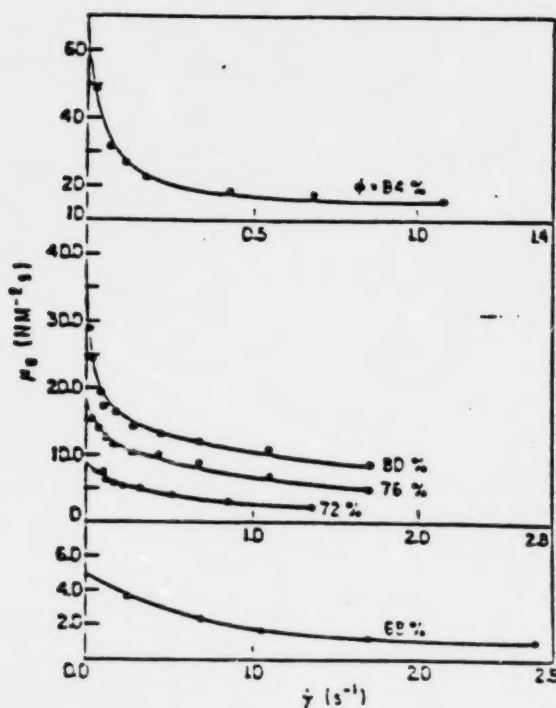


Fig. 2 Estimation of zero shear viscosity

RELATIONSHIP BETWEEN VISCOSITY AND OIL CONCENTRATION

For solid-liquid suspensions a number of empirical and semi-empirical relationships are available relating viscosity of suspensions to the volume concentration of solids. Chong and co-workers (1971) studied the concentration effect of neutrally buoyant spherical particles on suspension viscosity and proposed the following correlation to fit their data on glass bead - polyisobutylene suspension.

$$\mu_r = (1 + A \frac{\phi/\phi_m}{1 - \phi/\phi_m})^B \quad (3)$$

where $A = 0.75$ and $B = 2$ are two parameters and ϕ is the volume fraction. ϕ_m is the maximum volume fraction attainable and μ_r is the relative viscosity.

In case of non-Newtonian suspensions relative viscosities will have to be estimated on the basis of apparent viscosities. Ghosh and Bhattacharya (1982) in a recent study of coal-oil suspension rheology used zero shear viscosity to calculate relative viscosity. In this study relative viscosity has been estimated at 0, 100 and 400 sec^{-1} shear rates. In addition η has also been used to determine relative viscosity at high shear rate.

A plot of μ_r versus ϕ at different shear rates is shown in Fig. 3. The relationship shown in Fig. 3 is similar to the solid-liquid suspension behaviour in that at higher concentration, the viscosity increases dramatically and becomes almost asymptotic at a very high concentration. However, these relations are very much affected by the particle size and size distribution of oil particles in the emulsion which means for different batches of emulsions μ_r will be different at the same shear rate and ϕ .

By comparison of photomicrographs of different concentration emulsions prepared in this study, it was seen that the droplet size and distribution did not vary significantly with the disperse phase concentration. Variation in particle size is, however, seen for the same concentration emulsions having different emulsifier content. A typical photomicrograph is given in Fig. 4 showing the broad size range of particles present in the emulsion.

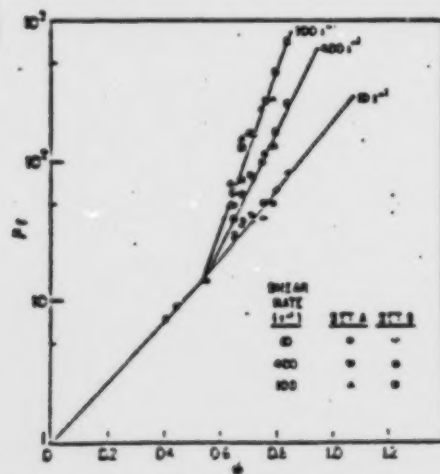


Fig. 3 Relative viscosity versus oil concentration

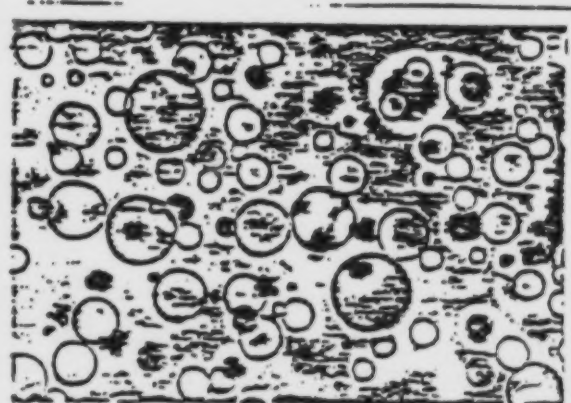


Fig. 4 Photomicrograph of 76% oil-in-water emulsion. Particle size range is 2 μm to 63 μm .

MAXIMUM VOLUME CONCENTRATION

Maximum volume concentration attainable in a dispersion may be influenced by many factors such as particle size, size distribution, shape, particulate surface charge, interaction between particles and medium, concentration of emulsifier etc. For solid-liquid suspensions, many workers have used ϕ_m and obtained correlations in terms of v_r and ϕ/ϕ_m . Recently Low and Bhattacharya (1983) have demonstrated that with the introduction of ϕ_m , v_r data for coal-oil suspensions of different moisture concentrations could be expressed by a single curve of v_r versus ϕ/ϕ_m . In this study also ϕ_m has been used to correlate all viscosity data. ϕ_m was determined from the experimental data of v_r and ϕ using Thomas's method (1965) of plotting $(v_r - 1)^{-1}$ versus ϕ and extrapolating the curve to a zero value of $(v_r - 1)^{-1}$. In Fig. 5 a plot of $(v_r - 1)^{-1}$ versus ϕ is shown where v_r has been given for different shear rates. It is interesting to see that extrapolations for various shear rates produce the same value for ϕ_m . In this case ϕ_m is estimated to be 0.905 for Set A and 0.89 for Set B. Experimentally it was observed that a stable emulsion of ϕ higher than 0.9 could not be prepared for Set A and for Set B the limit was 0.88. Beyond this concentration it was noted that a separate oil phase appeared in conjunction with the oil-in-water emulsion phase. It is presumed that emulsion becomes saturated and rather unstable near its maximum concentration where coalescence can easily work and therefore where this limit is exceeded there exist two phases namely oil and oil-in-water emulsion. This point of maximum concentration and instability may, therefore, be estimated from the method described.

RELATIVE VISCOSITY VERSUS REDUCED OIL CONCENTRATION

In Fig. 6 relative apparent viscosity is plotted against ϕ/ϕ_m as a function of shear rate. Data are presented for the shear rates of 0, 100 and 400 sec^{-1} respectively and high shear rate data have also been expressed in terms of n/v_c where n is the plastic viscosity given in Table 2. It is obvious from Fig. 6 that v_r versus ϕ/ϕ_m produces a series of curves within the limits of zero shear rate data and the plastic viscosity data.

The relative viscosity at zero shear rate is about two orders of magnitude higher than those corresponding to 100 sec^{-1} . This is because of the existence of large number of flocs of oil globules at zero shear rate. In Fig. 7, v_r at zero shear rate has been compared with the empirical expression of Ghosh and Bhattacharya (1982) for black coal-oil suspension as given below:

$$v_r = (1 + 0.163 \frac{\phi/\phi_m}{1 - \phi/\phi_m})^{9.3} \quad (4)$$

Comparison has also been made with the empirical expression for Chong and co-workers given in equation (3). It can be seen in Fig. 7 that v_r values for emulsions are much higher than those for the coal-oil suspensions for the same values of ϕ/ϕ_m . This may be due to the fact that flocculation is quite significant in case of emulsions, particularly at high dispersed phase concentrations, resulting in higher viscosities. The other contributing

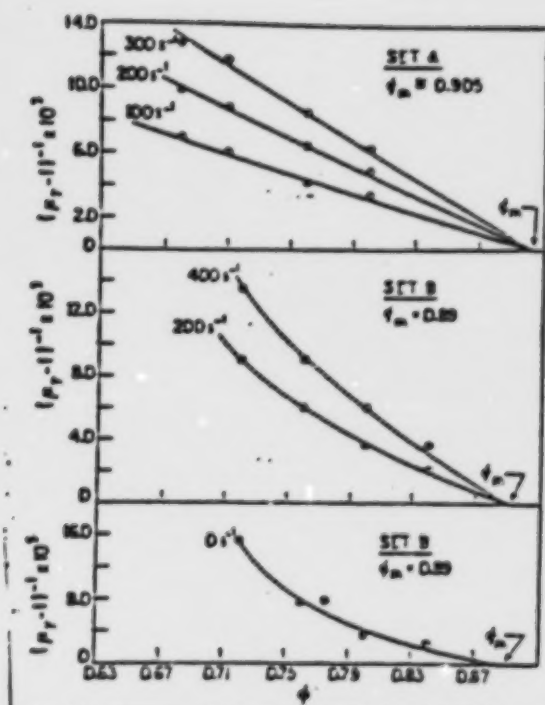


Fig. 5 Estimation of maximum attainable oil concentration



Fig. 6 Relative viscosity versus reduced oil concentration

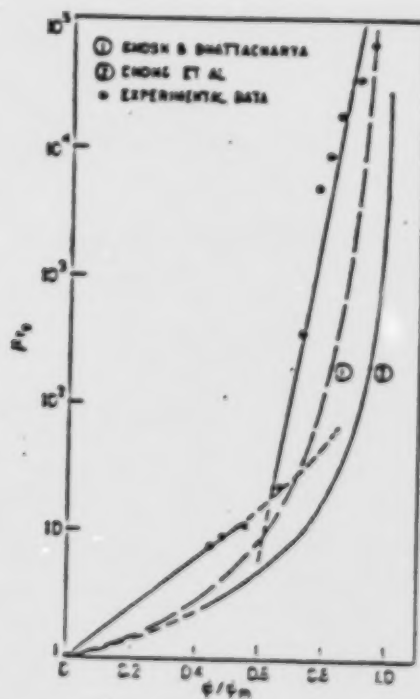


Fig. 7 Comparison of zero shear viscosity

factor could be that although oil globules in the emulsion might behave as rigid particles the interfacial viscosity, because of the presence of the emulsifier, will be much higher than the continuous phase viscosity thus giving rise to higher viscosity in case of emulsions. Coal-oil suspensions have been observed to agglomerate heavily at very high concentration. The data of Chong and co-workers as shown in Fig. 7 are much lower than the present work particularly at high concentrations. This is because Chong's expression is valid only for Newtonian non-interacting systems. Except for the data of Ghosh and Bhattacharya, no other data in literature were found to compare with the present emulsion data. The following empirical relationships were obtained relating zero shear rate viscosity and ϕ/ϕ_m :

$$\eta_r = 1 + 7.92 \left(\frac{\phi/\phi_m}{1 - \phi/\phi_m} \right)^{1.21} \quad (5)$$

for $0 \leq \phi/\phi_m \leq 0.66$

and

$$\eta_r = 2.4 \times 10^{-8} \exp (13.75 \phi/\phi_m) \quad (6)$$

for $\phi/\phi_m > 0.66$

CORRELATION FOR HIGH SHEAR VISCOSITY

The high shear data have been expressed by means of the Bingham Plastic relation as shown in equation (2). The plastic viscosity η , which is independent of shear rate is used to obtain the correlation. The relative plastic viscosity data (η_r vs ϕ/ϕ_m) from both Set A and Set B are found to be in good agreement. Regression analysis of the data gave the following best fit relationship:

$$\eta_r = 0.84 \exp (2.07 \phi/\phi_m) \quad (7)$$

However since equation (7) does not reduce to 1 when ϕ/ϕ_m equals zero an alternative relationship was also obtained as follows:

$$\eta_r = \exp (1.95 \phi/\phi_m) \quad (8)$$

where η_r becomes equal to 1 for ϕ/ϕ_m equal to zero. Equation (7) is found to fit the data more accurately, particularly for ϕ/ϕ_m greater than 0.6. There is no data available in literature to compare with these results.

SUMMARY

Mineral oil-in-water emulsions exhibit non-Newtonian behaviour for concentrations beyond 55 percent. At lower concentrations, they behave as

Newtonian fluids. Emulsion viscosity increases sharply with the increase in oil concentration.

The maximum attainable oil concentration in the emulsion estimated from the rheological data is in close agreement with the actual value of maximum oil concentration that could be achieved experimentally.

Based on experimental data, equations which correlate zero shear viscosity and infinite shear viscosity as a function of reduced oil concentration (ϕ/ϕ_m) have been proposed.

The emulsion appears to be unstable near its maximum attainable concentration. When the maximum concentration is exceeded, the emulsion separates into oil and emulsion phase.

ACKNOWLEDGEMENTS

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NOMENCLATURE

- A = constant in Equation 3, dimensionless
B = constant in Equation 3, dimensionless
k = power law constant, $\text{NM}^{-2} \text{s}^n$
n = flow behaviour index, dimensionless

$\frac{du}{dr}$ = shear rate, s^{-1}

Greek Letters

- η = plastic viscosity, $\text{NM}^{-2} \text{s}$
 η_r = relative plastic viscosity η/η_c , dimensionless
 ϕ = dispersed phase concentration, dimensionless
 ϕ_m = maximum attainable dispersed phase concentration, dimensionless
 $\dot{\gamma}$ = shear rate, s^{-1}
 η_a = apparent viscosity, $\text{NM}^{-2} \text{s}$
 η_{ah} = apparent viscosity at high shear rate, $\text{NM}^{-2} \text{s}$
 η_c = viscosity of continuous phase, $\text{NM}^{-2} \text{s}$
 η_r = relative viscosity η/η_c , dimensionless
 τ = shear stress, NM^{-2}
 τ_y = yield stress, NM^{-2}

PAPER 6

TITLE: A NOVEL VISCOSITY CORRELATION FOR NON-NEWTONIAN
CONCENTRATED EMULSIONS

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RUNNING TITLE: EMULSION VISCOSITY CORRELATION

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ABSTRACT

The flow properties of concentrated oil-in-water and water-in-oil emulsions have been determined by using the Fann coaxial cylinder viscometer, the Weissenberg Rheogoniometer and the Brookfield viscometer. Based on the extensive amount of experimental data collected, an empirical equation which correlates the relative viscosities of emulsions (both oil-in-water and water-in-oil) as a function of normalized dispersed phase concentration is proposed. According to the equation, if the viscosity of emulsion at one specific dispersed phase concentration is known, the viscosity at other concentrations of dispersed phase can be predicted. A comparison of the correlation has been made with those proposed for solid-in-liquid suspensions.

INTRODUCTION

The flow properties of concentrated emulsions are of interest in many applications. For example, the choice of mixing equipment and power requirements are dependent upon the rheological properties of the emulsion being mixed. The prediction of the transport behaviour of emulsions through pipelines (1-2) requires a knowledge of possible variations of viscosity with shearing rate as well as with concentration. Oil-well drilling (3), fracturing and acidizing processes (4) and all operations in which large volumes of concentrated emulsions are handled, require understanding and control of the viscous properties of concentrated emulsions. Despite the importance of the problem, there has been developed no commonly accepted theoretical or empirical expression (5) to correlate the viscosity of highly concentrated emulsions.

The development of an empirical correlation which correlates the relative viscosities of oil-in-water and water-in-oil emulsions as a function of dispersed phase concentration was the main objective of this study. Other objectives included:

1. the prediction of the viscosities of different emulsions using the correlation and comparison of the predicted values with the measured viscosities.
2. a comparison of the proposed viscosity correlation for emulsions with those published for solid-liquid suspensions.

EXPERIMENTAL WORK

Oils:

The oils used in this study were: Bayol-35, Bayol-35/ CCl_4 mixture, Shell Vitrea 22 and Shell Vitrea 220.

Bayol-35 is a refined white mineral oil. It has a density of 794 kg/m^3 at 15°C and viscosity of 2.3 cst at 40°C .

Shell Vitrea 22 and Shell Vitrea 220 are highly refined straight mineral paraffinic lubricating oils. Shell Vitrea 22 has a density of 858.2 kg/m^3 at 15°C and viscosity of about 22.5 cst at 40°C . Shell Vitrea 220 has a density of 881.7 kg/m^3 at 15°C and viscosity of about 210 cst at 40°C .

The Bayol-35/ CCl_4 mixture was made up of 73.3% by volume Bayol-35 and 26.7% by volume analytical grade carbontetrachloride.

The various emulsion systems studied were:

- a) Bayol-35/ CCl_4 -in-water emulsions at room temperature over a dispersed phase concentration range of 30% to 82%.
- b) Bayol-35-in-water emulsions at different temperatures over the dispersed phase concentration range of 40% to 69%.
- c) Shell Vitrea 220-in-water emulsions at different temperatures over a dispersed phase concentration range of 19% to 69%. The effect of aging was also studied for this system.
- d) Water-in-Bayol-35/ CCl_4 emulsions at room temperature over a dispersed phase concentration range of 0% to 75%.
- e) Water-in-Shell Vitrea 22 emulsions at room temperature over a dispersed phase concentration range of 50% to 60%.

Emulsifiers:

The commercially available surfactants TRITON X-100 and SPAN 80 were used to stabilize the oil-in-water and water-in-oil emulsions respectively. TRITON X-100 (iso-octylphenoxypolyethoxy ethanol) is a non-ionic water soluble emulsifier and SPAN 80 (sorbitan monooleate) is a non-ionic oil soluble emulsifier.

Emulsification Procedure:

The emulsions were prepared in approximately 900 ml batches. For systems other than Bayol-35/ CCl_4 , each concentration of emulsion was made fresh. However, for Bayol-35/ CCl_4 system, higher concentrations were achieved by adding more dispersed phase to lower-concentration emulsion.

In all cases, oil-in-water emulsions were prepared by the agent-in-water method (emulsifier dissolved in the water phase) and water-in-oil emulsions were prepared by the agent in oil method (emulsifier dissolved in the oil phase). To prepare water-in-oil emulsions, 1.5% by volume of SPAN 80 based on the oil phase was used. In case of Bayol-35/ CCl_4 oil-in-water emulsions, 1.0% by volume of Triton X-100 based on the water phase (deionized water) was used. For other oil-in-water emulsions (Shell Vitrea 220 and Bayol-35 emulsions), 0.5% by volume of Triton X-100 based on the water phase was employed.

A variable speed model 1-LV Gifford-Wood homogenizer was used to mix the oil and water. For each system, the speed and time of mixing was kept constant throughout the dispersed phase concentration range.

Viscometers Used:

For low viscosity emulsions (at lower concentrations), a Brookfield viscometer model LV with U.L. adapter was used.

For higher viscosity emulsions, most of the data were collected by use of the Fann coaxial cylinder viscometer model 35A. To check the reliability of this instrument, data for Bayol-35/cc₁, emulsions were also collected by use of the Weissenberg Rheogoniometer model R-18.

Up to about a dispersed phase concentration of 70%, the data obtained from the two instruments agreed very well. At concentrations greater than 70%, the Fann viscometer gave higher viscosities.

RESULTS AND DISCUSSION

At lower concentrations of dispersed phase (< 20%), the emulsions behaved like Newtonian fluids. At higher concentrations, the emulsions behaved like pseudoplastic fluids. The flow curves of shear stress versus shear rate were found to follow a linear relationship on a log-log plot. A power law model could be fitted to the data according to the equation:

$$\tau = K(\dot{\gamma})^n \quad [1]$$

The power law parameters determined were then used to obtain the viscosities at different shear rates.

At very high shear rates, the flow curves showed a linear relationship between the shear stress and the shear rate on a linear plot. A Bingham model was fitted to this data according to the equation:

$$\tau = \tau_y + \mu_p \dot{\gamma} \quad [2]$$

where μ_p , a plastic viscosity, is the slope of the linear portion of the flow curve and τ_y , a yield stress, is the intercept of the line drawn through the linear portion of the flow curve.

The factors (other than the shear rate) which affected the viscosity of the emulsions were: dispersed phase concentration, viscosity of the dispersed phase, temperature, aging and the type of emulsion (O/W or W/O).

The viscosity of emulsions was found to increase almost exponentially with the dispersed phase concentration.

To see the effect of viscosity of the dispersed phase on the viscosity of emulsion, a comparison of high shear rate (1020 s^{-1}) viscosity data of Bayol 35-in-water and shell vitrea 220-in-water emulsions was made. Shell vitrea 220 was about 100 times more viscous than Bayol 35. Variables such as the type and concentration of emulsifier, speed and time of mixing (which could all affect the viscosity of emulsions) were kept the same in both cases. The viscosity of shell vitrea 220 emulsions was found to be significantly higher than those of the Bayol emulsions.

On increasing the temperature, the viscosity of emulsions decreased significantly. The apparent viscosity versus the temperature data followed Arrhenius type relationship.

To see the effect of aging on the viscosity of emulsions, the high shear rate (1020 s^{-1}) viscosity data of fresh and aged (10 days) shell

vitrea 220 oil-in-water emulsions was compared. The viscosity of aged emulsions was found to be lower than those of the fresh emulsions.

On comparing the relative viscosity versus the dispersed phase concentration data of Bayol/ CCl_4 -in-water and water-in-Bayol/ CCl_4 emulsions (at 20 s^{-1}), water-in-oil emulsions were found to be more viscous than oil-in-water emulsions at the same ϕ .

DEVELOPMENT OF VISCOSITY CORRELATION

In order to correlate the viscosity data of emulsions as a function of dispersed phase concentration, a parameter which will be a function of all the possible factors affecting the viscosity has to be incorporated into the correlation.

For solid-liquid suspensions, several workers (6-7) have used ϕ_{max} to incorporate the effect of particle size and distribution in the correlation. ϕ_{max} is the maximum volume concentration of solid phase that can be achieved in the suspension. At ϕ_{max} or packed bed concentration of dispersed phase, the viscosity of the suspension becomes infinite. ϕ_{max} can be determined experimentally by plotting $1/\nu_r$ versus ϕ and extrapolating the data to the point where $1/\nu_r$ is zero. This ϕ_{max} will depend upon the particle size and size distribution of the dispersed phase. The suspension viscosity correlation proposed by Chong and co-workers (6) is:

$$\nu_r = \left[1 + 0.75 \frac{(\phi/\phi_{\text{max}})}{1 - (\phi/\phi_{\text{max}})} \right]^2 \quad [3]$$

A similar correlation was also proposed by Frankel and Acrivos (7):

$$\nu_r = \frac{9}{8} \left[\frac{(\phi/\phi_{\max})^{1/3}}{1 - (\phi/\phi_{\max})^{1/3}} \right] \quad [4]$$

In the case of emulsions, there is no well defined $\phi_{\max}$. The viscosity of emulsions is always finite although the rate of change of viscosity with concentration increases as the concentration increases. At very high concentrations of dispersed phase, there is the possibility of phase inversion (i.e. an o/w emulsion may invert to w/o emulsion and vice versa). The dispersed phase concentration at which this inversion occurs is usually uncertain. The mode of addition of dispersed phase is very critical. For example, at higher concentrations, if small amounts of dispersed phase are added, inversion will usually occur at very high concentrations. If larger amounts of dispersed phase are added, then inversion may occur at relatively lower concentrations. Furthermore, the phase inversion can involve an intermediate stage (8) where multiple phases (such as oil suspended in a continuous aqueous phase but with smaller water droplets suspended in the oil) are formed and this will make the calculation of the inversion point more uncertain.

In order to take into account the many factors which would affect the viscosity of emulsions, $(\phi)_{\mu_r=100}$ was arbitrarily chosen. $(\phi)_{\mu_r=100}$ is that concentration of dispersed phase at which the relative viscosity of the emulsion becomes 100. This concentration will be a function of all the possible factors which affect the viscosity of emulsions. For example, if the average particle size of an emulsion is lower, the relative viscosity will be higher and $(\phi)_{\mu_r=100}$ will be less.

To determine $(\phi)_{\mu_r=100}$, the viscosity data were plotted as $(1/\mu_r) \times 10^3$ versus ϕ on a semi-log plot (see Fig. 1 for a typical plot). The value of ϕ at which $(1/\mu_r) \times 10^3$ reached a value of 10 was taken as $(\phi)_{\mu_r=100}$. The plastic viscosity data for Shell Vitrea 220 oil-in-water emulsions was used to determine $(\phi)_{\mu_{pr}=100}$, where μ_{pr} is a relative plastic viscosity.

Figure 1 shows that different shear rates give different values of $(\phi)_{\mu_r=100}$. As the shear rate increases, the value of $(\phi)_{\mu_r=100}$ increases. This is because of the fact that at higher shear rates, the viscosities of the emulsions are lower and therefore a higher dispersed phase concentration is required to reach $\mu_r=100$. Table 1 summarizes the values of $(\phi)_{\mu_r=100}$ obtained for different systems at different shear rates.

Having determined the values of $(\phi)_{\mu_r=100}$, the viscosity data of non-Newtonian emulsions were plotted as μ_r versus $\phi/(\phi)_{\mu_r=100}$ on a semi-log plot. Figure 2 shows the plot of μ_r versus $\phi/(\phi)_{\mu_r=100}$. All the viscosity data for different systems at different shear rates fall on a single curve. The plastic viscosity data (μ_{pr} versus $\phi/(\phi)_{\mu_{pr}=100}$) also followed the same curve.

The data for Newtonian emulsions (i.e. lower dispersed phase concentrations) followed the same curve provided $(\phi)_{u_r=100}$ was used as the normalizing dispersed phase concentration.

The best fit curve through the data is represented by the following equation.

$$u_r = \left[1 + \frac{1.342 \phi / (\phi)_{u_r=100}}{1.194 - \phi / (\phi)_{u_r=100}} \right]^{2.226} \quad [5]$$

In order to compare this result with the viscosity correlations proposed for suspensions (Eqs. [3] and [4]), $\phi_{\max}$ was determined in terms of $(\phi)_{u_r=100}$ by substituting a value of $u_r=100$ into these correlations and the following new relationships (for suspensions) between u_r and $\phi / (\phi)_{u_r=100}$ were obtained:

For Chong and co-workers correlation:

$$\phi_{\max} = \frac{13}{12} (\phi)_{u_r=100} \quad [6]$$

∴ Chong's model reduces to:

$$u_r = \left[1 + \frac{0.75 \phi / (\phi)_{u_r=100}}{13/12 - \phi / (\phi)_{u_r=100}} \right]^2 \quad [7]$$

For Frankel and Acrivos correlation:

$$\phi_{\max} = 1.0341311 (\phi)_{u_r=100} \quad [8]$$

∴ Frankel and Acrivos model reduces to:

$$u_r = \frac{9}{8} \left[\frac{(\phi / (\phi)_{u_r=100})^{1/3}}{1.01125 - (\phi / (\phi)_{u_r=100})^{1/3}} \right] \quad [9]$$

A comparison of all three correlations is made in Figure 3. All the correlations pass through the point where $\mu_r=100$ and $\phi/(\phi)_{\mu_r=100} = 1$. The present correlation exceeds the predictions of the solid-liquid suspension ones. The displacement of the emulsion data above the solid suspension data is possibly related to aggregation and deformation of the emulsion particles. These effects are not considered in suspension correlations. However, an exact explanation is not known at this time. An interesting point to note is that at concentrations where $\phi/(\phi)_{\mu_r=100}$ approaches values slightly greater than 1.0, the solid-liquid suspension correlations give infinite viscosities whereas the present correlation gives finite viscosities. This is because of the fact that emulsion viscosity will always remain finite due to the deformable nature of the dispersed phase whereas suspensions will have infinite viscosities as packed bed concentrations (ϕ_{max}) are approached.

PREDICTION OF VISCOSITY OF EMULSIONS FROM THE PROPOSED CORRELATION

In order to predict the viscosity of emulsion over a wide range of dispersed phase concentration, viscosity versus the shear rate data at one specific concentration is required. Using the values of known relative viscosity, at a given shear rate, and known dispersed phase concentration we can determine $(\phi)_{\mu_r=100}$ from the correlation. For any other dispersed phase concentration (in the non-Newtonian range), $\phi/(\phi)_{\mu_r=100}$ can be calculated and this value when substituted in the correlation will give relative viscosity at the shear rate under consideration. This procedure can then be repeated to calculate relative viscosities at other shear rates.

To use the correlation for Newtonian emulsions (i.e. at lower dispersed phase concentrations), the relative plastic viscosity of any one specific concentration non-Newtonian emulsion is required. Using the relative plastic viscosity and the dispersed phase concentration we can determine $(\phi)_{\mu_{pr}=100}$ from the correlation. For any other dispersed concentration (in the Newtonian range), $\phi/(\phi)_{\mu_{pr}=100}$ can be calculated and this value when substituted in the correlation will give the predicted relative viscosity.

The correlation has been tested for the following systems:

- (a) Water-in-oil emulsions where oil phase was Shell Vitrea 22.
- (b) Aged oil-in-water emulsions where oil phase was Shell Vitrea 220.
- (c) Heavy oil-in-water emulsions where oil phase was cold lake heavy crude oil. The data for this system was taken from the published literature (9).

For Shell Vitrea 22 w/o emulsions, $(\phi)_{\mu_r=100}$ at different shear rates was determined by using the correlation and the viscosity data of emulsion having a dispersed phase concentration of 60%. Knowing $(\phi)_{\mu_r=100}$, $\phi/(\phi)_{\mu_r=100}$ was calculated for other concentrations (50% and 55%) at different shear rates and these values were then substituted in the correlation to calculate the predicted μ_r values.

For aged Shell Vitrea 220 o/w emulsions, $(\phi)_{\nu_r=100}$ at different shear rates was determined by using the viscosity data of 68.6% emulsion. The relative viscosity ν_r , was then predicted for other concentrations (65.13%, 62%, 59% and 53.85%) at different shear rates by using the correlation.

For heavy oil-in-water emulsions, $(\phi)_{\nu_r=100}$ at a shear rate of 1000 s^{-1} was determined by using the viscosity data of 71.2% emulsion. Knowing $(\phi)_{\nu_r=100}$, ν_r at 1000 s^{-1} was predicted for other concentrations in the range 40% - 71.2% by using the correlation.

Figure 4 shows the comparison between the predicted viscosity values and the true (measured) viscosities. There is excellent agreement between the predicted and the measured values and in most cases the deviation of the predicted from the measured values is less than 6%.

CONCLUSIONS:

Based on the extensive amount of data, an equation which correlates the relative viscosities of both o/w and w/o emulsions as a function of normalized dispersed phase concentration is proposed. Comparison of the proposed correlation with those published for solid-liquid suspensions show that for $0.30 < \phi/(\phi)_{\nu_r=100} < 1.0$, the proposed correlation gives higher viscosities.

The proposed correlation can be used to predict the viscosity of emulsion over a wide range of dispersed phase concentration provided the viscosity of emulsion at one specific concentration is known. The predicted and the measured values of viscosities for three emulsion test systems are in close agreement.

ACKNOWLEDGEMENTS:

Appreciation is expressed to CANMET for a research contract to enable this investigation to proceed.

NOMENCLATURE

- K = consistency index, $\text{NM}^{-2} \text{s}^{-n}$
 n = flow behaviour index, dimensionless
o/w = oil-in-water emulsion
w/o = water-in-oil emulsion

Greek Letters

- ϕ = ratio of dispersed phase volume to the total volume, dimensionless
 $\dot{\gamma}$ = shear rate, s^{-1}
 ν_r = relative viscosity, dimensionless
 τ = shear stress, NM^{-2}

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Table 1: Values of $(\epsilon)_{\nu_r = 100}$ for different systems

Type of system	Shear rate (s^{-1})	$(\epsilon)_{\nu_r = 100}$
Bayol-35 oil-in-water emulsions (T = 27°C)	50	0.70
	100	0.72
	150	0.725
	520	0.7725
	1020	0.80
Bayol-35 oil-in-water emulsions-- (T = 35°C)	50	0.6925
	100	0.705
	150	0.722
	520	0.772
	1020	0.813
Bayol-35 oil-in-water emulsions (T = 64°C)	50	0.675
	100	0.702
	150	0.71
	520	0.755
	1020	0.788
Shell vitrea 220 oil-in-water emulsions (T = 27°C)	10	0.675
	50	0.695
	150	0.705
	520	0.72
	1020	0.73
Bayol-35/ CCl_4 water-in-oil emulsions (T = 25°C)	10	0.66
	50	0.695
	520	0.77
	1020	0.802

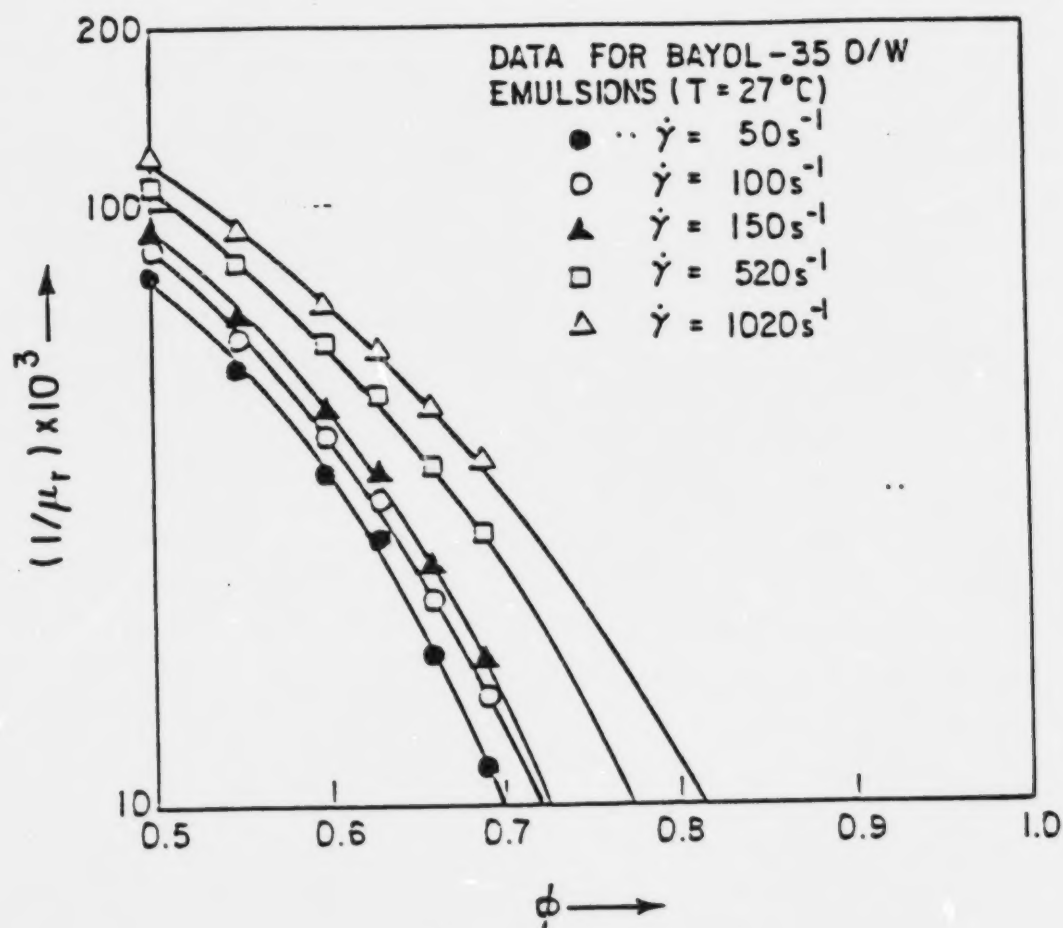


Figure 1 Estimation of $(\phi)_{v_r = 100}$

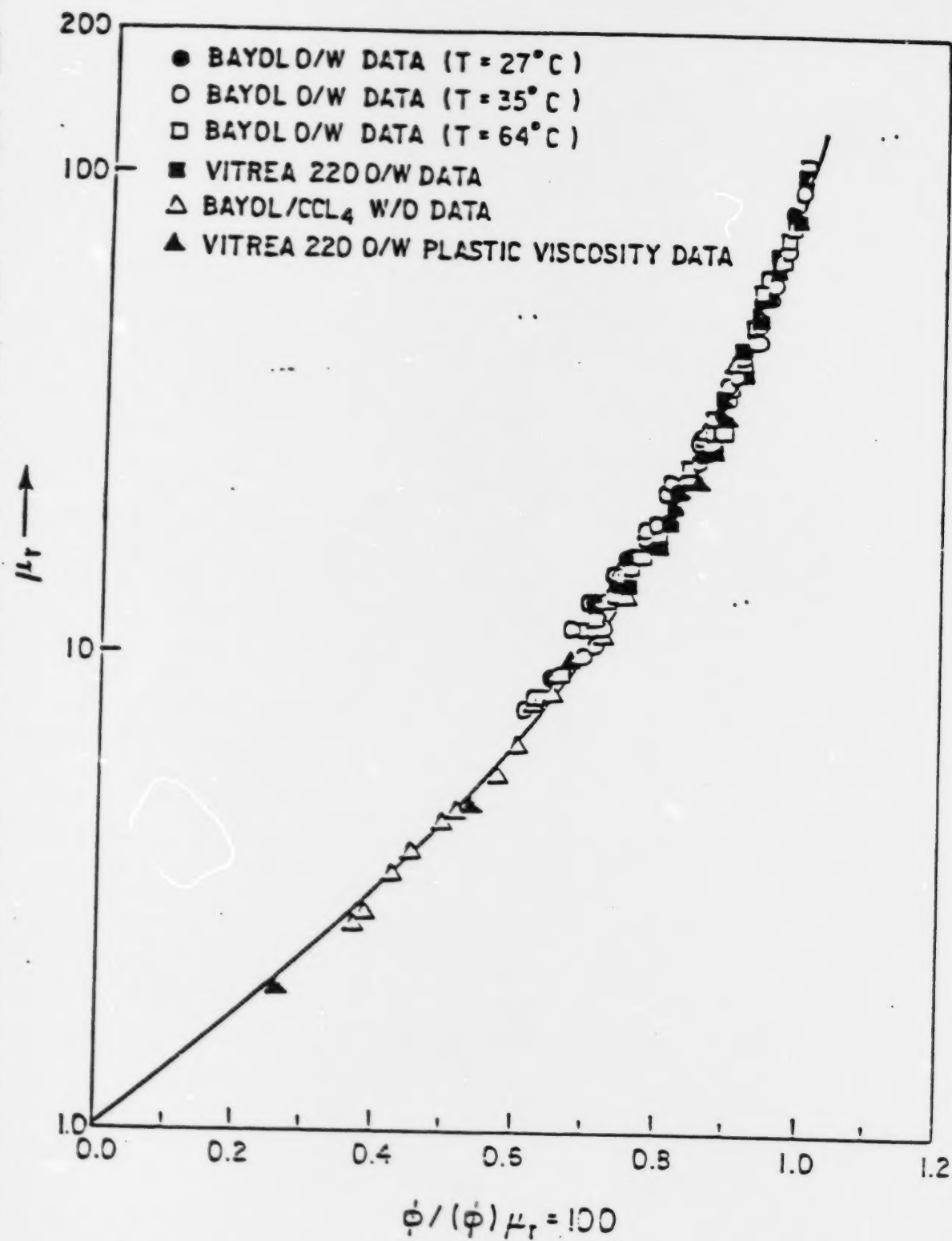


Figure 2 Relative viscosity versus
 the normalized concentration

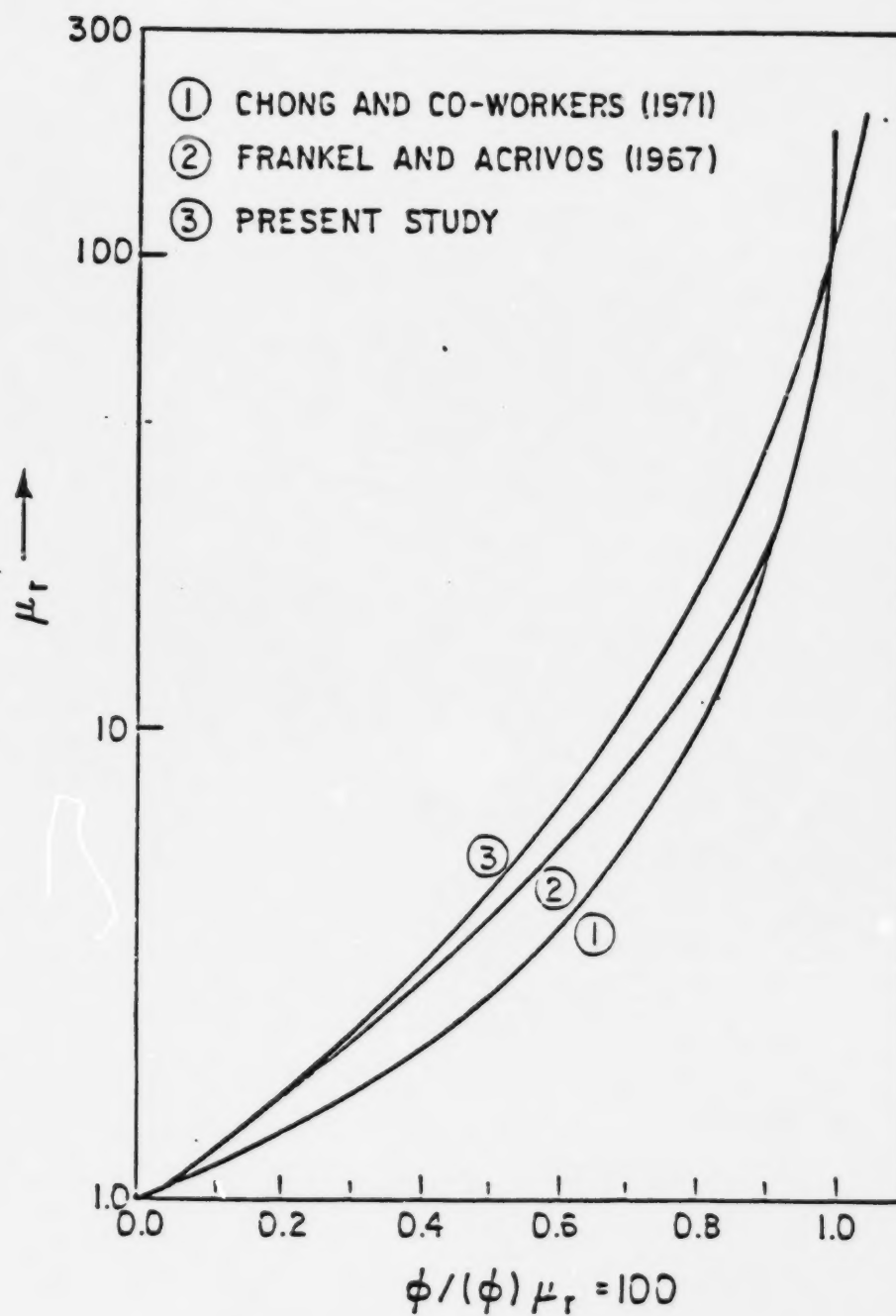


Figure 3 Comparison of suspension viscosity models with the present emulsion viscosity model.

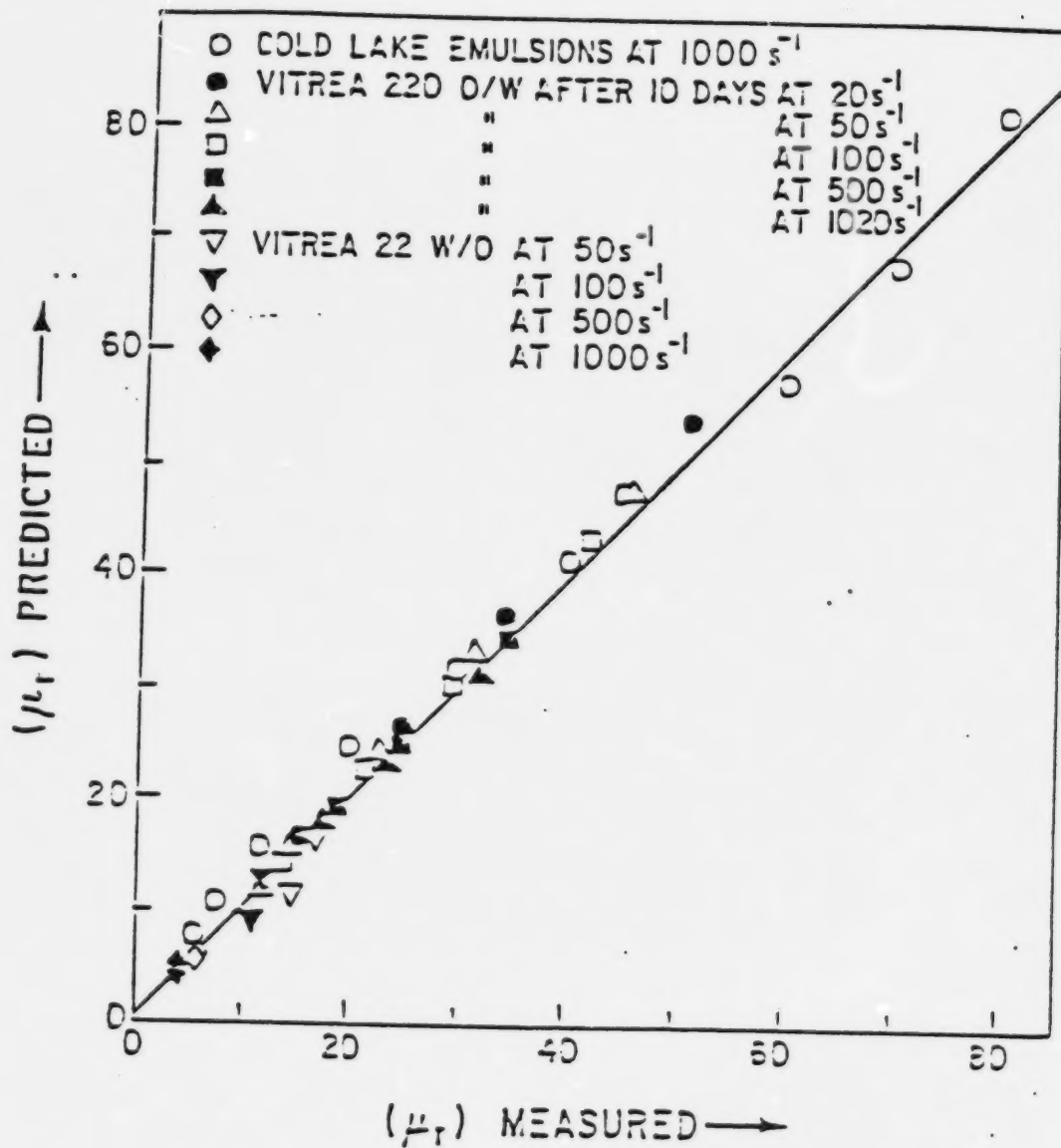


Figure 4 Predicted relative viscosity versus the measured relative viscosity

PAPER 6b

TITLE: VISCOSITY CORRELATION FOR NON-NEWTONIAN
CONCENTRATED EMULSIONS

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ABSTRACT

The Rheological behaviour of concentrated oil-in-water and water-in-oil emulsions has been investigated by using the Fann coaxial cylinder viscometer, the Weissenberg Rheogoniometer and the Brookfield viscometer. The effects of aging, viscosity of dispersed phase and temperature on the rheology of emulsions were determined. Based on the extensive amount of experimental data collected, an empirical equation which correlates the relative viscosities of emulsions (both oil-in-water and water-in-oil) as a function of normalized dispersed phase concentrations is proposed. The equation takes into account all possible factors which affect the viscosity of emulsions such as particle size and distribution, viscosity of dispersed phase, type of emulsion, nature and concentration of emulsifying agent etc. According to the equation, if the rheology of one (specific concentration) emulsion is known, the rheology of other concentrations of emulsions can be predicted. A comparison of the proposed correlation has been made with those proposed for solid-in-liquid suspensions.

INTRODUCTION

The flow properties of concentrated emulsions are of interest in many applications. For example, the choice of mixing equipment and power requirements are dependent upon the rheological properties of the emulsion being mixed. The prediction of the transport behaviour of such materials through pipelines (1-2) requires a knowledge of possible variations of viscosity with shearing rate as well as with concentration. Oil-well drilling (3), fracturing and acidizing processes (4) and all operations in which large volumes of concentrated emulsions are handled, such as emulsion drilling fluids, emulsion pad fluids and emulsified acids, require understanding and control of the viscous properties of concentrated emulsions. Despite the importance of the problem, there has been developed no commonly accepted theoretical or empirical expression to correlate the viscosity of highly concentrated emulsions.

The development of an empirical correlation which correlates the relative viscosities of oil-in-water and water-in-oil emulsions as a function of dispersed phase concentration was the main objective of this study. Other objectives included:

1. the determination of the effect of aging on the rheology.
2. the determination of the effect of viscosity of dispersed phase on the rheology of oil-in-water emulsions.
3. the determination of the effect of temperature on the rheology.
4. the prediction of the viscosities of different emulsions using the correlation and comparison of the predicted values with the measured viscosities.
5. a comparison of the proposed viscosity correlation for emulsions with those published for solid-liquid suspensions.

PREVIOUS WORK:

The viscosity of an emulsion depends upon several factors such as:

1. The viscosity of the external phase.
2. The viscosity of the internal phase.
3. The relative volumes of the two phases.
4. The nature and the concentration of the emulsifying agent.
5. The average particle size and particle size distribution.
6. Temperature.

For relatively dilute emulsions, considerable theoretical and empirical work has been done in correlating the viscosity with some of the variables mentioned above. For suspensions of non-deformable spherical globules at low concentrations Einstein (6, 7) derived the following relationship between the viscosity of the suspension and the volume fraction:

$$\nu_r = \frac{\nu}{\nu_c} = 1 + 2.5 \phi \quad [1]$$

The assumptions made in arriving at Eq. [1] were: no interaction between the globules and no slippage at the interface.

Most emulsions of practical interest exceed the concentration for which Eq. [1] is valid. With increasing concentration, the interaction between the globules increases and eventually mechanical interference occurs between the particles as packed bed concentrations are approached.

To take into account the increased hydrodynamic interaction, many investigators (8-13) have expanded Eq. [1] into the polynomial form;

$$\mu_r = 1 + k_1 \phi + k_2 \phi^2 + k_3 \phi^3 \quad [2]$$

and determined the constants that would fit their data.

Based on the analogy with the influence of variable pressure on a material which obeys Hooke's law, Richardson (14) calculated the compressibility of an emulsion whose dispersed phase volume concentration is increased from ϕ to $\phi + \Delta\phi$. From this he derived the following expression for the relative viscosity of concentrated emulsions at any rate of shear.

$$\ln \mu_r = k_R \phi \quad [3]$$

where k_R is a constant. Broughton and Squires (15) found that Eq. [3] did not give values of μ_r which agreed with their experimental data, so they amended it to achieve more satisfactory agreement,

$$\ln \mu_r = k_R \phi + Y_R \quad [4]$$

where Y_R is an additional constant.

Hatschek (16) found that when ϕ exceeded 0.5 the linear portion of the shear stress versus the shear rate curve could be represented by

$$\mu_r = \frac{1}{(1-\phi^{1/3})} \quad [5]$$

Sibree (17, 18) obtained higher values than those predicted by Eq. [5]. He attributed the discrepancy to an increase in the size of the particles due to hydration of the absorbed emulsifier layer by continuous phase fluid. This was considered to increase ϕ by a factor h_s , so Eq. [5] was amended to:

$$\mu_r = \frac{1}{1 - (h_s \phi)^{1/3}} \quad [6]$$

The main drawback to all the relations proposed so far is that the constants are very system dependent and are strong functions of shear rate, temperature, average particle size and particle size distribution, temperature, viscosity of internal phase and nature of emulsifying agent etc. Even if all these variables are kept constant, the existing relations still fail to fit a broad spectrum of experimental data.

EXPERIMENTAL WORK

Oils:

The oils used in this study were: Bayol-35, Bayol-35/cc₄, mixture, Shell Vitrea 22 and Shell Vitrea 220.

Bayol-35 is a refined white mineral oil. It has a density of 794 kg/m³ at 15°C and viscosity of 2.3 cst at 40°C.

Shell Vitrea 22 and Shell Vitrea 220 are highly refined straight mineral paraffinic lubricating oils. Shell Vitrea 22 has a density of 858.2 kg/m³ at 15°C and viscosity of about 22.5 cst at 40°C. Shell Vitrea 220 has a density of 881.7 kg/m³ at 15°C and viscosity of about 210 cst at 40°C.

The Bayol-35/ CCl_4 mixture was made up of 73.3% by volume Bayol-35 and 26.7% by volume analytical grade carbontetrachloride.

The various emulsion systems studied were:

- a) Bayol-35/ CCl_4 -in-water emulsions at room temperature over a dispersed phase concentration range of 30% to 82%.
- b) Bayol-35-in-water emulsions at different temperatures over the dispersed phase concentration range of 40% to 69%.
- c) Shell Vitrea 220-in-water emulsions at different temperatures over a dispersed phase concentration range of 19% to 69%. The effect of aging was also studied for this system.
- d) Water-in-Bayol-35/ CCl_4 emulsions at room temperature over a dispersed phase concentration range of 0% to 75%.
- e) Water-in-Shell Vitrea 22 emulsions at room temperature over a dispersed phase concentration range of 50% to 60%.

Emulsifiers:

The commercially available surfactants TRITON X-100 and SPAN 80 were used to stabilize the oil-in-water and water-in-oil emulsions respectively. TRITON X-100 (iso-octylphenoxy polyethoxy ethanol) is a non-ionic water soluble emulsifier and SPAN 80 (sorbitan monooleate) is a non-ionic oil soluble emulsifier.

Emulsification Procedure:

The emulsions were prepared in approximately 900 ml batches. For systems other than Bayol-35/ CCl_4 , each concentration of emulsion was made fresh. However, for Bayol-35/ CCl_4 system, higher concentrations were achieved by adding more dispersed phase to lower-concentration emulsion.

In all cases, oil-in-water emulsions were prepared by the agent-in-water method (emulsifier dissolved in the water phase) and water-in-oil

emulsions were prepared by the agent in oil method (emulsifier dissolved in the oil phase). To prepare water-in-oil emulsions, 1.5% by volume of SPAN 80 based on the oil phase was used. SPAN 80 was found to be readily soluble in all the oils studied. In case of Bayol-35/ CCl_4 oil-in-water emulsions, 1.0% by volume of Triton X-100 based on the water phase (deionized water) was used. For other oil-in-water emulsions (Shell Vitrea 220 and Bayol-35 emulsions), 0.5% by volume of Triton X-100 based on the water phase was employed. Triton X-100 was found to be readily soluble in the water phase.

A variable speed model 1-LV Gifford-Wood homogenizer was used to mix the oil and water. For each system, the speed and time of mixing was kept constant throughout the dispersed phase concentration range.

Viscometers Used:

Fann viscometer: The Fann coaxial cylinder viscometer model 35A having 6 test speeds (3, 6, 100, 200, 300 and 600 r.p.m.) and an electrically heated sample cup with precision temperature control was used in this study. The outer stainless steel cylinder (R-1 rotor) had a radius of 1.8415 cm. Except for Shell Vitrea 22 water-in-oil emulsions, only one inner cylinder (B-1 bob) having a radius of 1.7245 cm and a height of 3.8 cm was used. Therefore, for the R-1/B-1 system, the gap width was 1.17 mm. For Shell Vitrea 22 water-in-oil emulsions, three different size bobs (B-1, B-2 and B-3) were used. B-2 and B-3 had radii of 1.2276 and 0.8622 cm respectively. The height of all the bobs was the same (3.8 cm).

Weissenberg rheogoniometer: This instrument was used only for Bayol-35/ CCl_4 oil-in-water and water-in-oil emulsions. The model used in this study was R-18. The platen diameter was 100 mm and cone angle was 4° .

giving a clearance between the cone flat (truncated tip) and adjacent plate of 171 μm .

Brookfield viscometer: For low viscosity emulsions, a Brookfield synchro-lectric viscometer model LV with U.L. adapter was used. The U.L. adapter consists of a precision cylindrical spindle rotating inside an accurately machined tube. This instrument gives the direct reading of viscosity rather than the shear stress versus the shear rate measurement. Therefore, this instrument was used only when the emulsion flow behaviour was Newtonian.

Calibration: Before collection of any experimental data, all the instruments were calibrated and the necessary calibration constants were determined. The Brookfield viscosity standards of 8.7 cp, 100 cp and 1000 cp (at 25°C) were used to calibrate the instruments.

RESULTS AND DISCUSSION

At lower concentrations of dispersed phase (< 20%), the emulsions behaved like Newtonian fluids. At higher concentrations, the emulsions behaved like pseudoplastic fluids. The flow curves of shear stress versus shear rate were found to have a linear relationship on a log-log plot. A power law model could be fitted to the data according to the equation:

$$\tau = K(\dot{\gamma})^n \quad [7]$$

At very high shear rates, the flow curves showed a linear relationship between the shear stress and the shear rate on a linear plot. A Bingham model was fitted to this data according to the equation:

$$\tau = \tau_y + \mu_p \dot{\gamma} \quad [8]$$

where μ_p , a plastic viscosity, is the slope of the linear portion of the flow curve and τ_y , a yield stress, is the intercept of the line drawn through the linear portion of the flow curve.

A typical set of flow curves obtained by using different types of instruments for water-in-oil emulsions, where the oil phase is a mixture of Bayol-35 and CCl_4 , is shown in Figure 1. The dashed lines for 0%, 10% and 20% emulsions (which were Newtonian) indicate the viscosity measurements obtained from a Brookfield U.L. adapter. Since this instrument gave only the viscosity of the emulsion (i.e. not shear stress versus shear rate), the dashed lines were drawn by using Newton's law of viscosity. The data points on the 10% and 20% emulsion lines were obtained from the Weissenberg Rheogoniometer. The solid lines shown in the figure are drawn through the Weissenberg Rheogoniometer data. Data obtained from the different instruments agree very well for dispersed phase concentrations lower than 70%. At 70% and higher concentrations, significant deviation is seen between the data from Fann and Weissenberg viscometers. The deviations in flow curves for the same emulsion are due to the existence of abnormal "wall effects" at the surface of cone-and-plate viscometer (20). Due to preferential migration of dispersed phase particles away from the walls, a low viscosity region is created adjacent to the surfaces. Major shearing occurs in this region and different viscometers give different emulsion

viscosity. Similar behaviour was also seen in the case of oil-in-water emulsions (Figure 2). Data from different instruments agreed only when the dispersed phase concentrations were lower than 70%. At higher concentrations, significant deviations were seen. Due to the existence of these abnormal wall effects at higher concentrations, the higher concentration data for the system under discussion were ignored for correlation purposes. For other systems, data were collected only up to a 70% dispersed phase concentration with the help of the Fann viscometer. Using a power law model, k and n were determined for each emulsion. This model was then used to determine the viscosities at different shear rates. A typical set of k and n values for water-in-Bayol/ CCl_4 emulsions is given in Table 1.

The relationship between relative viscosity and dispersed phase concentration depends upon several factors such as:

- (i) shear rate
- (ii) particle size and distribution
- (iii) viscosity of dispersed phase.
- (iv) nature and concentration of emulsifying agent.
- (v) type of emulsion (o/w or w/o?)
- (vi) Temperature
- (vii) Flow properties of continuous phase. (If the continuous phase is non-Newtonian, then even very dilute emulsions will behave like non-Newtonian fluids.)

In the following sections, we will discuss the effects of some of the above mentioned factors:

Effect Of Shear Rate On The Viscosity Of Emulsions:

As discussed in the previous section, most of concentrated emulsions ($\phi > 20\%$) behave like pseudoplastic fluids and the viscosity decreases as the shear rate is increased. It is a well known fact (21) that at high dispersed phase concentrations, the dispersed particles in emulsions tend to flocculate (due to the presence of London-Van der Waals' attraction forces) and in this process, the particles entrap some of the continuous phase fluid which then becomes part of the dispersed phase. Therefore, the dispersed phase to continuous phase ratio is increased and viscosity also increases as compared to unflocculated same-concentration emulsion. As the shear rate is increased, the flocs break up and continuous phase fluid is released. Thus the effective volume ratio of dispersed phase to continuous phase decreases and this leads to a decrease in viscosity. When the shear rates become very high, most of the flocs break and effective volume ratio corresponds to the true value, provided no solvation effects are involved.

Figure 3 shows a typical plot of relative viscosity versus concentration with shear rate as a parameter. This plot has been obtained from Bayol-in-water emulsion data. Decrease in viscosity with shear rate is higher in the initial shear rate range. This is due to the fact that more flocs (to be broken) are available in the initial shear range.

Particle Size and Distribution:

The non-Newtonian nature of emulsions is mainly due to the formation of aggregates of dispersed particles. The other factor which could lead to non-Newtonian behaviour is the "solvation" or "hydration" effect. Here, the effective volumes of the dispersed particles increase due to hydration of the emulsifier layer around them. The magnitude of both flocculation and hydration effects is highly dependent upon the particle size and

distribution. In general, wider size distributions and larger average particle sizes give lower viscosities than narrow size distributions and smaller particle sizes.

Figure 4 shows a plot of viscosity versus dispersed phase concentration (at shear rate of 1020s^{-1}) for fresh and aged (10 days) Shell Vitrea 220 oil-in-water emulsions (aging of emulsions was done at room temperature in air tight bottles). The viscosities of fresh emulsions are higher than those of the aged emulsions. On aging, coalescence of particles occurs to form larger particles and therefore the average particle size increases with age thus reducing the viscosity. The globule size distribution is also affected and it usually becomes broader (22) on aging.

Viscosity Of Dispersed Phase:

Unlike solid-in-liquid suspensions, the dispersed phase in emulsions is a deformable fluid. The transmission of tangential and normal stresses from the continuous phase to the dispersed phase can occur and this leads to fluid circulation within the globules, which, in turn reduces the distortion of flow patterns around the globules. Reduction of distortion of flow pattern leads to lower emulsion viscosities as compared to solid-in-liquid suspensions for the same concentration of dispersed phase. Internal circulation of fluid is dependent upon the viscosity of dispersed phase. As the viscosity of disperse phase increases, internal circulation decreases and therefore the viscosity of emulsion tends to increase.

The particle size and distribution also depends upon the viscosity of dispersed phase. Keeping all other factors constant, if the viscosity of dispersed phase is increased, the average particle size will probably increase. This will lead to a decrease in the viscosity of emulsion.

The third factor which could effect the viscosity of the emulsion is the deformation of the dispersed particles. The more the deformation of the dispersed particles, the operator is the viscosity of emulsion.

To determine the effect of viscosity of the dispersed phase, a comparison of rheological characteristics of Bayol-in-water and Shell Vitrea 220-in-water emulsions was made. The viscosity of shell vitrea 220 was about 100 times the viscosity of Bayol-35 at 40°C. In both these systems, other variables such as the nature and concentration of emulsifier, speed and time of shearing (which could all affect the viscosity of the emulsions) were kept constant. The Bayol-in-water emulsions were found to be much more non-Newtonian than the Shell Vitrea 220 emulsions. Tables II(a) and II(b) list the values of power law constants for both of these systems.

The higher degree of non-Newtonian behaviour in the Bayol emulsions as compared to the Shell Vitrea 220 emulsions is probably because of the different chemical constitution of the internal phase. The chemical constitution of the internal phase can affect configuration at the oil-water interface which may in turn, lead to different degrees of non-Newtonian behaviour.

At lower shear rates, the viscosities of the Bayol emulsions were significantly higher than those of the Shell Vitrea 220 because of the higher degree of non-Newtonian behaviour. However, at higher shear rates, the viscosities of the Bayol emulsions were lower than those of the Shell Vitrea 220 emulsions. Figure 5 shows the plot of relative viscosity versus concentration at $1020s^{-1}$ for both systems. At concentrations greater than about 40%, the viscosity of the Bayol emulsions is significantly lower. This is probably because the internal circulation within the oil

globules in case of Shell Vitrea 220 emulsions is inhibited due to higher oil viscosity. At lower concentrations ($<40\%$), the viscosities of both emulsion systems (at shear rate of $1020s^{-1}$) are similar. Since the deformation of particles lead to higher emulsion viscosities and internal circulation leads to lower emulsion viscosities, it is possible that at lower concentrations deformation of particles in the case of Bayol emulsions is significant as compared to the internal circulation effect. Therefore, the viscosity of Bayol emulsions is high and comparable to Shell Vitrea 220 emulsions.

Effect of Temperature:

The effect of temperature on the rheology of emulsions was determined only for the case of Shell Vitrea 220-in-water and Bayol-in-water emulsions. Rheological data was collected at four different temperatures ($27^{\circ}C$ - $78^{\circ}C$). The samples were heated in a special electrically heated thermocup provided with a Fann viscometer. The sample was kept in the cup, at the desired temperature, for about 10 minutes to ensure equilibrium.

In case of Bayol-35 oil-in-water emulsions, the power law consistency index, K , was found to decrease significantly with temperature. The flow behaviour index, n , did not vary significantly. Table III(a) shows the values of K and n at different temperatures (for different concentration emulsions) for Bayol-35 emulsions.

For Shell Vitrea 220 oil-in-water emulsions, the flow behaviour index, n , decreased as temperatures were increased to $64^{\circ}C$. A slight increase in n was seen at temperature greater than $64^{\circ}C$. The power law index, K , did not show any trend. Table III(b) shows the values of K and n at different temperatures for Shell Vitrea 220 emulsions. The decrease in n with temperature is probably because of increased flocculation of dispersed

particles with temperature. However, at temperatures greater than 64°C, migration of emulsifier away from the interface could become significant and thus would lead to more coalescence of particles to form bigger globules. This, in turn, would increase the flow behaviour index.

When the apparent viscosity (at a particular shear rate) for any emulsion is plotted as a function of $1/T$ (Figure 6), a linear relationship is observed. The data can be fitted by Arrhenius type relationship:

$$\eta_a = A_T \exp \left(\frac{E}{RT} \right) \quad [9]$$

where E is the molar activation energy for flow, R is the gas constant and T is the absolute temperature. The constant A_T is characteristic of fluid.

Tables IV(a) and IV(b) show the values of E/R and A_T at various shear rates for different concentration emulsions. In calculating these values, data of 78°C were not included. For both Shell Vitrea 220 and Bayol-35 emulsions, A_T decreases and E/R increases with the increase in shear rate at any value of ϕ .

For Bayol-35 emulsions, A_T increases with the increase in ϕ at any shear rate. At shear rates of 520 s^{-1} and 1020 s^{-1} , E/R is found to decrease with the increase in ϕ . At other shear rates (50 s^{-1} and 100 s^{-1}) also, there is a decrease in E/R with ϕ but there is more scatter of data.

For Shell Vitrea 220 emulsions, A_T increases with the increase in ϕ at shear rates of 50 s^{-1} and 100 s^{-1} . For shear rates of 520 s^{-1} and 1020 s^{-1} , A_T does not show any trend with ϕ . ϵ/R does not show any trend with ϕ at shear rates of 50 s^{-1} and 100 s^{-1} . However, at shear rates of 520 s^{-1} and 1020 s^{-1} , ϵ/R increases with ϕ .

Type of Emulsions:

Figure 7 shows a plot of relative viscosity versus dispersed phase concentration (at 20 s^{-1}) for both oil-in-water and water-in-oil emulsions. For any concentration, the relative viscosity of water-in-oil emulsions is more than the corresponding relative viscosity of oil-in-water emulsions. This could be due to various reasons. Firstly, the emulsifiers used to stabilize the two types of emulsions were different. For water-in-oil emulsions, SPAN 80 was used whereas TRITON x-100 was used to stabilize oil-in-water emulsions. Secondly, the average particle size and distribution could be different in the two cases which would lead to different viscosities. The deformative nature of the dispersed phase is also different in the two types of emulsions.

DEVELOPMENT OF VISCOSITY CORRELATION

As has been seen, the viscosity of emulsions is dependent upon several factors discussed in the previous sections. In order to correlate the viscosity data of emulsions as a function of dispersed phase concentration by a single correlation, a parameter which will be a function of all the possible factors affecting the viscosity has to be incorporated in the correlation.

For solid-liquid suspensions, several workers (23-24) have used $\phi_{\max}$ to incorporate the effect of particle size and distribution in the correlation. $\phi_{\max}$ is the maximum volume concentration of solid phase that can be achieved in the suspension. At $\phi_{\max}$ or packed bed concentration of dispersed phase, the viscosity of suspension becomes infinite. $\phi_{\max}$ can be determined experimentally by plotting $1/\nu_r$ versus ϕ and extrapolating the data to the point where $1/\nu_r$ is zero. This $\phi_{\max}$ will depend upon the particle size and distribution of the dispersed phase. The suspension viscosity model proposed by Chong and co-workers (23) is:

$$\nu_r = \left[1 + 0.75 \frac{(\phi/\phi_{\max})}{1 - (\phi/\phi_{\max})} \right]^2 \quad [10]$$

A similar model was also proposed by Frankel and Acrivos (24):

$$\nu_r = \frac{9}{8} \left[\frac{(\phi/\phi_{\max})^{1/3}}{1 - (\phi/\phi_{\max})^{1/3}} \right] \quad [11]$$

In the case of emulsions, there is no $\phi_{\max}$. The viscosity of emulsions is always finite although the rate of change of viscosity with concentration increases as the concentration increases. At very high concentrations of dispersed phase, there is a possibility of phase inversion (i.e. o/w emulsion may invert to w/o emulsion and vice versa). The dispersed phase concentration at which this inversion occurs is usually uncertain. The mode of addition of disperse phase is very critical. For example, at higher concentrations, if small amounts of dispersed phase are added, inversion will usually occur at very high concentrations. If larger

amounts of dispersed phase are added, then inversion may occur at lower concentrations.

Therefore, in order to take into account the factors which would affect the viscosity of emulsions, $(\phi)_{\mu_r=100}$ was chosen arbitrarily. $(\phi)_{\mu_r=100}$ is that concentration of dispersed phase at which the relative viscosity of the emulsion becomes 100. This concentration will depend upon all the possible factors which would affect the viscosity of emulsions. For example, if the average particle size of emulsions is lower, the relative viscosity will be more and $(\phi)_{\mu_r=100}$ will be less. It is a well known fact (5) that at very low concentrations of dispersed phase, particle size effects are not important. At very high concentrations also where there is mechanical interference between particles, particle size effects will probably be less significant. To avoid both these situations, it was decided to choose $(\phi)_{\mu_r=100}$.

To determine $(\phi)_{\mu_r=100}$, the viscosity data were plotted as $(1/\mu_r) \times 10^3$ versus ϕ on a semi-log plot (see Fig. 8 for typical plot). The value of ϕ at which $(1/\mu_r) \times 10^3$ reached a value of 10 was taken as $(\phi)_{\mu_r=100}$. The plastic viscosity data for Shell Vitrea 220 oil-in-water emulsions was used to determine $(\phi)_{\mu_{pr}}$, where μ_{pr} is a relative plastic viscosity.

Figure 8 shows that different shear rates give different values of $(\phi)_{\mu_r=100}$. As the shear rate increases, the value of $(\phi)_{\mu_r=100}$ increases. This is because of the fact that at higher shear rates, the viscosities of the emulsions are lower and therefore a higher dispersed phase concentration is required to reach $\mu_r=100$. Table V summarizes the values of $(\phi)_{\mu_r=100}$ obtained for different systems at different shear rates.

Having determined the values of $(\phi)_{\mu_r=100}$, all the viscosity data were plotted as μ_r versus $\phi/(\phi)_{\mu_r=100}$ on a semi-log plot. Figure 9 shows the plot of μ_r versus $\phi/(\phi)_{\mu_r=100}$. All the viscosity data for different systems at different shear rates fall on a single curve. The plastic viscosity data (relative plastic viscosity versus $\phi/(\phi)_{\mu_{pr}=100}$) also followed the same curve.

The data for Newtonian emulsions (i.e. lower dispersed phase concentrations) followed the same curve provided $(\phi)_{\mu_{pr}=100}$ was used as the normalizing dispersed phase concentration.

The best fit curve through the data is represented by the following equation.

$$\mu_r = \left(\frac{1.342 \phi/(\phi)_{\mu_r=100}}{1.194 - \phi/(\phi)_{\mu_r=100}} \right)^{2.226} \quad [12]$$

In order to compare our model with the viscosity models proposed for solid-liquid suspensions (Eqs. [10] and [11]) a value of $\mu_r=100$ was substituted in the Eqs. [10] and [11] and the relationships between $\phi_{\max}$ and $(\phi)_{\mu_r=100}$ were determined.

For Chong and co-workers model:

$$\phi_{\max} = \frac{13}{12} (\phi)_{\mu_r=100} \quad [13]$$

∴ Chong's model reduces to:

$$\mu_r = \left(1 + \frac{0.75 \phi/(\phi)_{\mu_r=100}}{13/12 - \phi/(\phi)_{\mu_r=100}} \right)^2 \quad [14]$$

For Frankel and Acrivos model:

$$\phi_{\max} \approx 1.0341311 (\phi)_{\nu_r=100} \quad [15]$$

∴ Frankel and Acrivos model reduces to:

$$\nu_r = \frac{9}{8} \left[\frac{\{\phi/(\phi)_{\nu_r=100}\}^{1/3}}{1.01125 - \{\phi/(\phi)_{\nu_r=100}\}^{1/3}} \right] \quad [16]$$

A comparison of all these models is made in Figure 10. All these models pass through the point where $\nu_r=100$ and $\phi/(\phi)_{\nu_r=100} \approx 1.0$. At lower concentrations, the Frankel and Acrivos model and the present model give similar viscosities. At higher concentrations, the Frankel and Acrivos model predicts lower viscosities. This may be because their model was based solely on the theory of hydrodynamic interactions of neighbouring spheres. Particle rotation, collision between particles and flocculation effects have been neglected in their model which could be very important in case of emulsions. Also, the deformation nature of emulsion dispersed phase could lead to higher viscosities. Chong et al's model also predicts lower viscosities than the present model. An interesting point to note is that at concentrations where $\phi/(\phi)_{\nu_r=100}$ approaches values slightly greater than 1.0, the solid-liquid suspension models give infinite viscosities whereas the present model gives finite viscosities. This is because of the fact that emulsion viscosity will always remain finite due to the deformative nature of the dispersed phase whereas suspensions will give infinite viscosities as packed bed concentrations ($\phi_{\max}$) are approached.

PREDICTION OF VISCOSITY OF EMULSIONS FROM THE PROPOSED CORRELATION

In order to predict the viscosity of emulsions over a wide ranges of concentrations, the rheology (viscosity versus shear rate) of one concentration of emulsion is required. Knowing the relative viscosity, at a particular shear rate, and concentration (ϕ), one can determine $(\phi)\mu_r=100$ from the correlation at that particular shear rate. Once $(\phi)\mu_r=100$ is known, one can then determine μ_r from the correlation for other concentrations at the shear rate under consideration. This procedure can then be repeated for other shear rates. It is recommended that the concentration of the emulsion for which the rheology is to be determined experimentally should be in the range $50\% < \phi < 70\%$.

To use the correlation for Newtonian emulsions (i.e. at lower dispersed phase concentrations), one should know the relative plastic viscosity for any one concentration non-Newtonian emulsion and use this viscosity to determine $(\phi)\mu_r=100$ from the correlation. Knowing $(\phi)\mu_r=100$, the value of ϕ for which the relative plastic viscosity is 100, one can determine the viscosity of Newtonian emulsions at other concentrations by using the correlation.

Since it was not possible to get reproducible data for concentrations greater than 75% because of the presence of "slip effects", the correlation is not tested for ϕ greater than 75%. However for lower concentrations, the correlation has been tested for the following systems:

- (a) Water-in-oil emulsions where oil phase was Shell Vitrea 22.
- (b) Aged oil-in-water emulsions where oil phase was Shell Vitrea 220.
- (c) Heavy oil-in-water emulsions where oil phase was cold lake heavy crude oil. The data for this system was taken from the published literature (25).

For Shell Vitrea 22 w/o emulsions, $(\phi)_{\mu_r} = 100$ at different shear rates was determined from 60% emulsion by using the correlation. Knowing $(\phi)_{\mu_r=100}$, μ_r was predicted from the correlation for other concentrations (50% and 55%) at different shear rates.

For aged Shell Vitrea 220 o/w emulsions, $(\phi)_{\mu_r=100}$ at different shear rates was determined from 68.6% emulsion by using the correlation. Knowing $(\phi)_{\mu_r=100}$, μ_r was predicted from the correlation for other concentrations (65.13%, 62%, 59% and 53.85%) at different shear rates.

For heavy oil-in-water emulsions, $(\phi)_{\mu_r=100}$ at a shear rate of 1000 s^{-1} was determined from 71.2% emulsion. Knowing $(\phi)_{\mu_r=100}$, μ_r at 1000 s^{-1} was predicted from the correlation for other concentrations in the range 40% - 71.2%.

Figure 11 shows the comparison between the predicted viscosity values and the true (measured) viscosities. There is excellent agreement between the predicted and the measured values. In most cases the deviation of the predicted values from the true values is within 6%.

CONCLUSIONS:

Based on the experimental data, the following conclusions can be made:

- (i) Emulsions behave like Newtonian fluids at lower dispersed phase concentrations. At higher concentrations, emulsions behave like power law fluids.
- (ii) The relationship between the relative viscosity and the dispersed phase concentration is non-linear. The rate of change of μ_r with ϕ increases with the increase in ϕ .

- (iii) The relationship of μ_r versus ϕ depends upon shear rate, average particle size and particle size distribution, viscosity of dispersed phase, type of emulsion, nature and concentration of emulsifying agents etc.
- (iv) The apparent viscosity of emulsion decreases with the increase in temperature. The temperature dependence of apparent viscosity can be expressed in the form of Arrhenius type law.
- (v) Highly concentrated emulsions (both oil-in-water and water-in-oil type) exhibit abnormal wall effects in the Weissenberg Rheogoniometer.
- (vi) Based on the extensive amount of data, an equation which correlates the relative viscosities of both o/w and w/o emulsions as a function of normalized dispersed phase concentration is proposed.
- (vii) Comparison of the proposed correlation with those published for solid-liquid suspensions show that for $0.30 < \phi/(\phi)_{\mu_r = 100} < 1.0$, the proposed correlation gives higher viscosities.
- (viii) There is no well defined $\phi_{\max}$ (maximum attainable dispersed phase concentration) in case of emulsions. However, for suspensions there is a well defined $\phi_{\max}$ which will depend upon the average particle size and particle size distribution. The viscosity of solid-in-liquid suspensions become infinite as $\phi_{\max}$ is approached.
- (ix) The proposed correlation can be used to predict the viscosities of emulsions provided the rheology of emulsion at one concentration is known. The predicted and the measured values of viscosities for three different emulsion systems tested are in close agreement.

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NOMENCLATURE

- K = consistency index, $\text{NM}^{-2} \text{s}^n$
n = flow behaviour index, dimensionless
o/w = oil-in-water emulsion
w/o = water-in-oil emulsion

Greek Letters

- ϕ = ratio of dispersed phase volume to the total volume, dimensionless
 γ = shear rate, s^{-1}
 μ = viscosity, Pas
 μ_c = viscosity of continuous phase, Pas
 μ_r = relative viscosity, dimensionless
 τ = shear stress, NM^{-2}

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Table I: Power law constants for Bayol-35/cc₂,
water-in-oil emulsions (T-25°C)

ϕ	n	$K(NM^{-2} s^{-n})$
0	1.0	2.01×10^{-3}
0.10	1.0	2.57×10^{-3}
0.20	1.0	3.92×10^{-3}
0.30	0.927	9.26×10^{-3}
0.40	0.922	1.54×10^{-2}
0.50	0.886	3.5×10^{-2}
0.60	0.788	13.7×10^{-2}
0.70	0.42	4.13

Table II(a): Power law constants for Bayol-35
oil-in-water emulsions (T=27°C)

ϕ	n	$K(NM^{-2} s^{-n})$
0.55	0.828	3.1×10^{-2}
0.60	0.785	5.57×10^{-2}
0.63	0.758	8.21×10^{-2}
0.66	0.69	17.0×10^{-2}
0.69	0.61	34.84×10^{-2}

Table II(b): Power law constants for Shell Vitrea
220 oil-in-water emulsions (T=27°C)

ϕ	n	$K(NM^{-2} s^{-n})$
0.5385	0.951	1.7×10^{-2}
0.59	0.93	2.79×10^{-2}
0.62	0.907	4.16×10^{-2}
0.6513	0.867	7.57×10^{-2}
0.686	0.845	13.74×10^{-2}

Table III(a): Power law constants for Bayol-35 oil-in-water emulsions at different temperatures

ϕ	Temp ($^{\circ}\text{C}$)	n	$K(\text{NM}^{-2}\text{s}^{-n})$
0.55	27	0.828	3.1×10^{-2}
	35	0.835	2.63×10^{-2}
	64	0.822	1.79×10^{-2}
	78	0.858	1.19×10^{-2}
0.60	27	0.785	5.57×10^{-2}
	35	0.786	4.9×10^{-2}
	64	0.787	3.09×10^{-2}
	78	0.786	2.69×10^{-2}
0.63	27	0.758	8.21×10^{-2}
	35	0.746	7.85×10^{-2}
	64	0.703	6.74×10^{-3}
	78	0.737	4.56×10^{-2}
0.66	27	0.69	17.0×10^{-2}
	35	0.676	16.39×10^{-2}
	64	0.628	14.71×10^{-2}
	78	0.656	11.2×10^{-2}
0.69	27	0.61	34.84×10^{-2}
	35	0.60	33.4×10^{-2}
	64	0.595	23.8×10^{-2}
	78	0.652	14.37×10^{-2}

Table III(b): Power law constants for shell vitrea 220
oil-in-water emulsions at different temperatures

ϕ	Temp. ($^{\circ}\text{C}$)	n	K ($\text{NM}^{-2} \text{s}^n$)
0.5385	27	0.951	1.7×10^{-2}
	36	0.91	1.77×10^{-2}
	64	0.907	1.16×10^{-2}
	78	0.926	9.03×10^{-3}
0.59	27	0.93	2.79×10^{-2}
	36	0.92	2.31×10^{-2}
	64	0.82	2.84×10^{-2}
	78	0.876	1.74×10^{-2}
0.62	27	0.907	4.16×10^{-2}
	36	0.856	4.59×10^{-2}
	64	0.772	4.73×10^{-2}
	78	0.754	4.82×10^{-2}
0.6513	27	0.867	7.57×10^{-2}
	36	0.83	7.67×10^{-2}
	64	0.695	10.26×10^{-2}
	78	0.731	7.51×10^{-2}
0.686	27	0.844	13.74×10^{-2}
	64	0.657	19.22×10^{-2}
	78	0.678	14.72×10^{-2}

Table IV(a): Arrhenius parameters for Bayol-35
oil-in-water emulsions

ϕ	shear rate (s^{-1})	A_T (Pas)	ϵ/R ($^{\circ}K$)
0.5	50	5.75×10^{-5}	1570
	100	4.58×10^{-5}	1610
	520	2.56×10^{-5}	1714
	1020	2.06×10^{-5}	1752
0.55	50	7.91×10^{-5}	1594
	100	6.7×10^{-5}	1609
	520	4.4×10^{-5}	1652
	1020	3.68×10^{-5}	1672
0.60	50	1.13×10^{-4}	1613
	100	9.8×10^{-5}	1610
	520	7.18×10^{-5}	1597
	1020	6.21×10^{-5}	1597
0.63	50	6.78×10^{-4}	1156
	100	4.01×10^{-4}	1263
	520	1.16×10^{-4}	1516
	1020	6.98×10^{-5}	1619
0.66	50	1.68×10^{-3}	1017
	100	9.3×10^{-4}	1129
	520	2.27×10^{-4}	1397
	1020	1.28×10^{-4}	1505
0.69	50	1.35×10^{-3}	1211
	100	9.48×10^{-4}	1235
	520	4.13×10^{-4}	1289
	1020	2.95×10^{-4}	1311

Table IV(b): Arrhenius parameters for shell vitrea 220
oil-in-water emulsions

ϕ	shear rate (s^{-1})	A_T (Pas)	ϵ/R ($^{\circ}K$)
0.59	50	5.23×10^{-4}	1097
	100	2.36×10^{-4}	1322
	520	3.56×10^{-5}	1860
	1020	1.64×10^{-5}	2081
0.62	50	7.78×10^{-4}	1086
	100	3.22×10^{-4}	1329
	520	3.94×10^{-5}	1911
	1020	1.67×10^{-5}	2149
0.6513	50	1.68×10^{-3}	983
	100	5.09×10^{-4}	1315
	520	2.97×10^{-5}	2105
	1020	9.3×10^{-6}	2428
0.686	50	2.46×10^{-3}	1019
	100	7.24×10^{-4}	1352
	520	3.93×10^{-5}	2145
	1020	1.19×10^{-5}	2470

Table V: Values of $(\phi)_{\mu_r = 100}$ for different systems

Type of system	Shear rate (s^{-1})	$(\phi)_{\mu_r = 100}$
Bayol-35 oil-in-water emulsions ($T = 27^\circ C$)	50	0.70
	100	0.72
	150	0.725
	520	0.7725
	1020	0.80
Bayol-35 oil-in-water emulsions ($T = 35^\circ C$)	50	0.6925
	100	0.705
	150	0.722
	520	0.772
	1020	0.813
Bayol-35 oil-in-water emulsions ($T = 64^\circ C$)	50	0.675
	100	0.702
	150	0.71
	520	0.755
	1020	0.788
Shell vitrea 220 oil-in-water emulsions ($T = 27^\circ C$)	10	0.675
	50	0.695
	150	0.705
	520	0.72
	1020	0.73
Bayol-35/ CCl_4 water-in-oil emulsions ($T = 25^\circ C$)	10	0.66
	50	0.695
	520	0.77
	1020	0.802

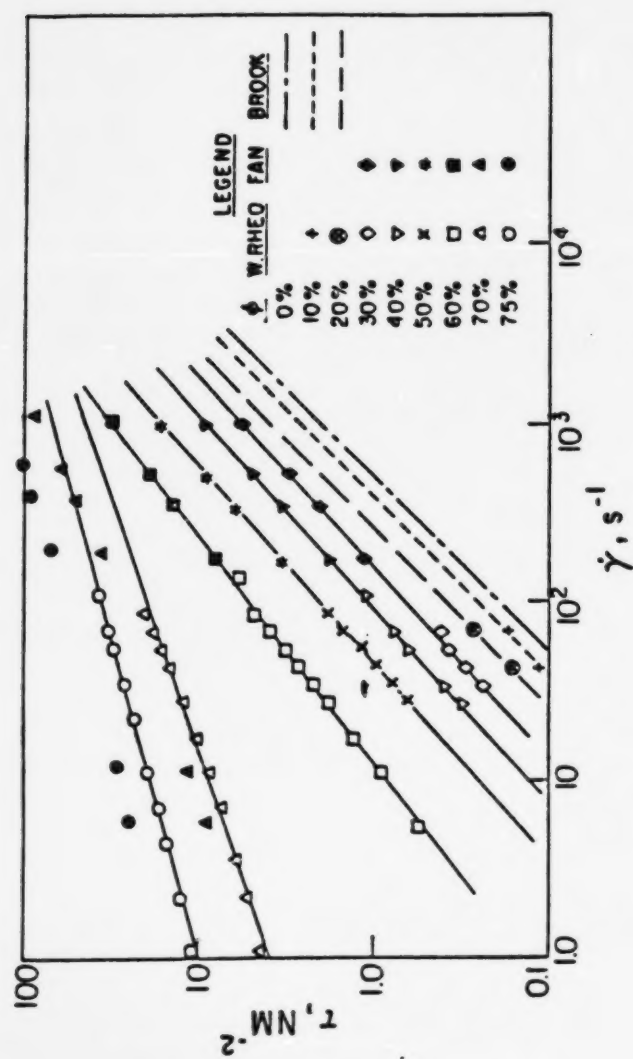


Figure 1: flow curves for water-in-oil emulsions

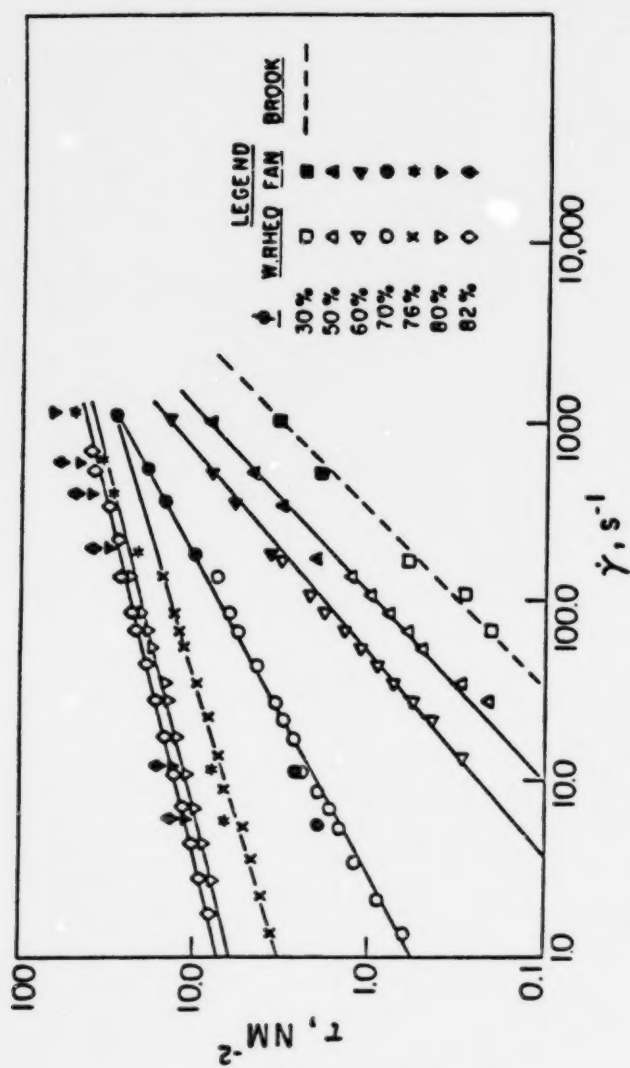


Figure 2: Flow curves for oil-in-water emulsions

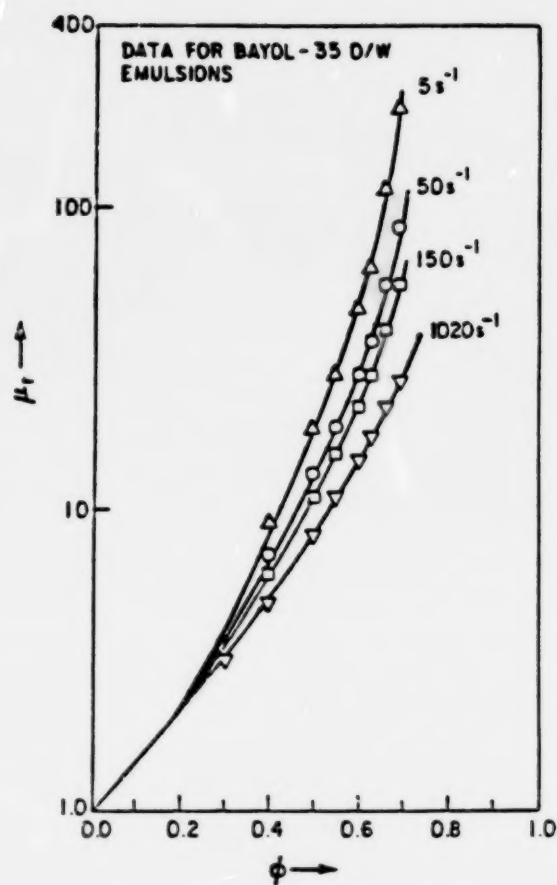


Figure 3: Effect of shear rate on the viscosity of emulsions

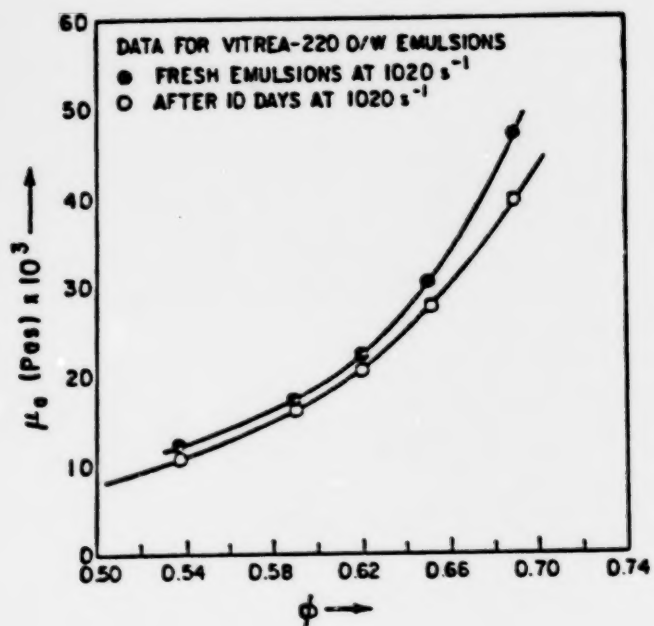


Figure 4: Effect of aging on the viscosity of emulsions

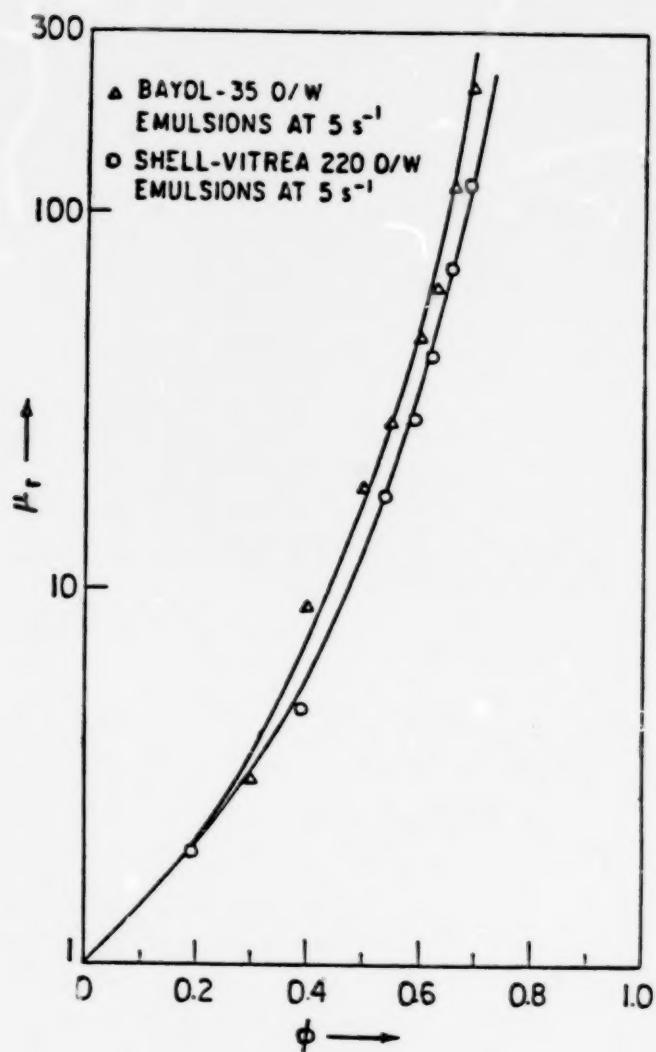


Figure 5: Comparison of Bayol-35 and Shell Vitrea 220 oil-in-water emulsions at low shear rates

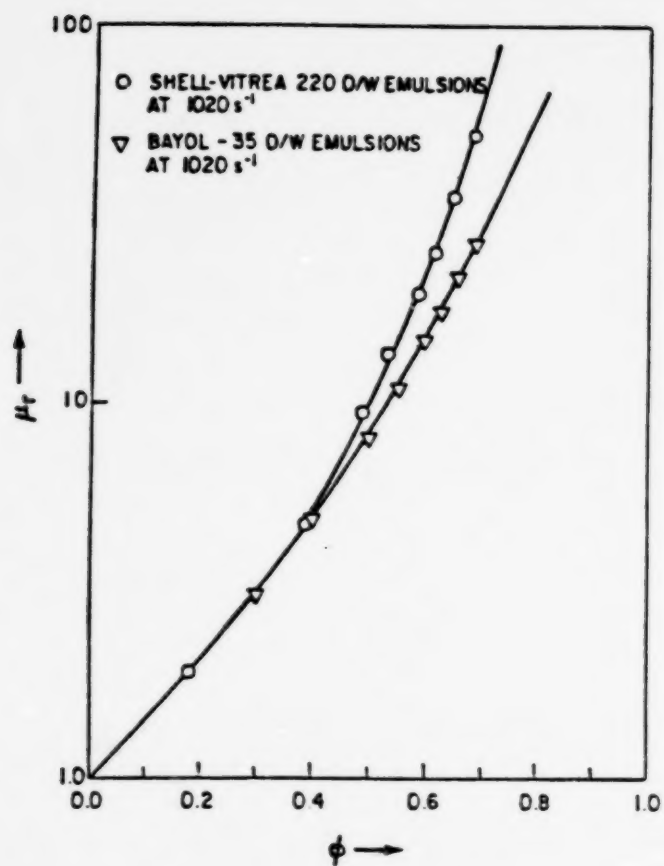


Figure 6: Comparison of Bayol-35 and Shell Vitrea 220 oil-in-water emulsions at high shear rates

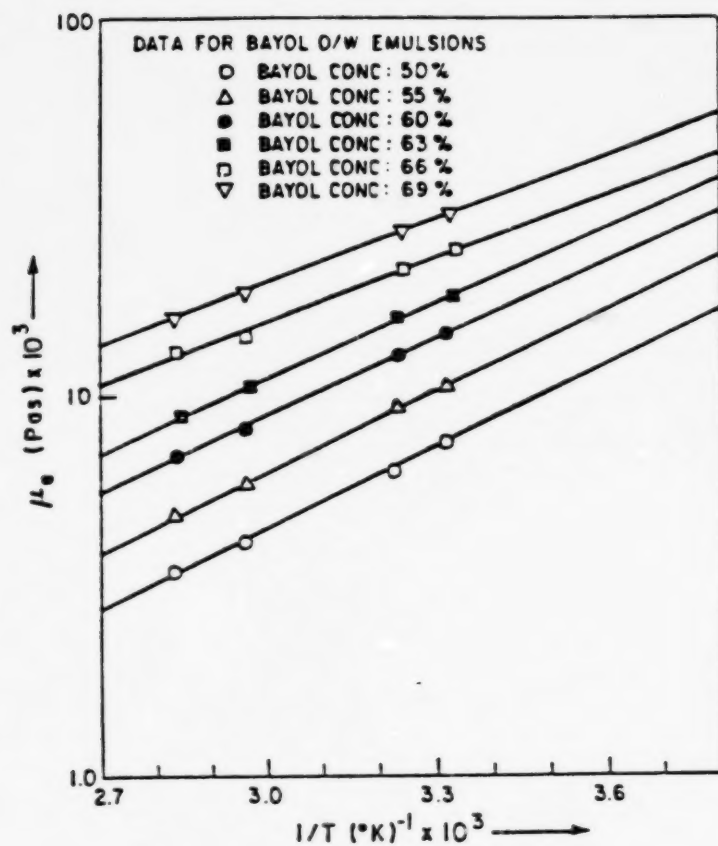


Figure 7: Effect of temperature on the viscosity of emulsions

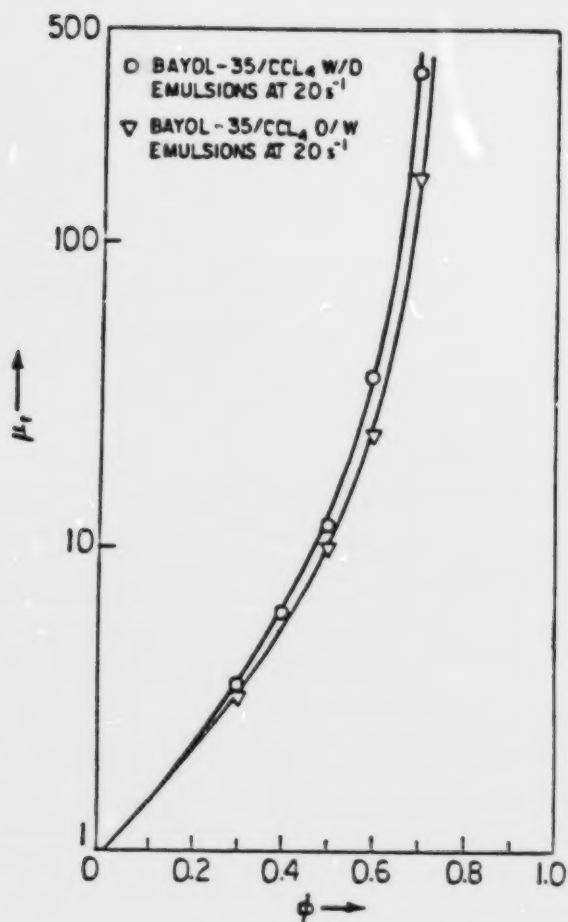


Figure 2: Comparison of water-in-oil and oil-in-water emulsions (oil phase: mixture of Bayol-35 and CCl_4)

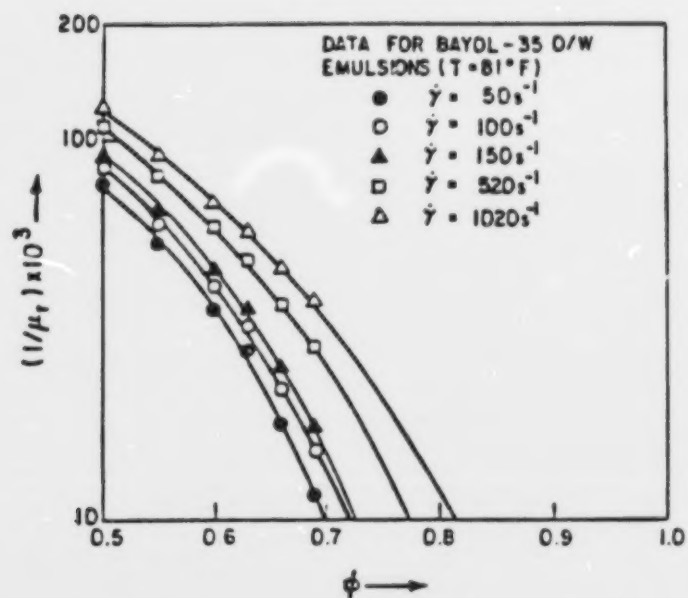


Figure 9: Estimation of $(c)_{\mu_r=100}$

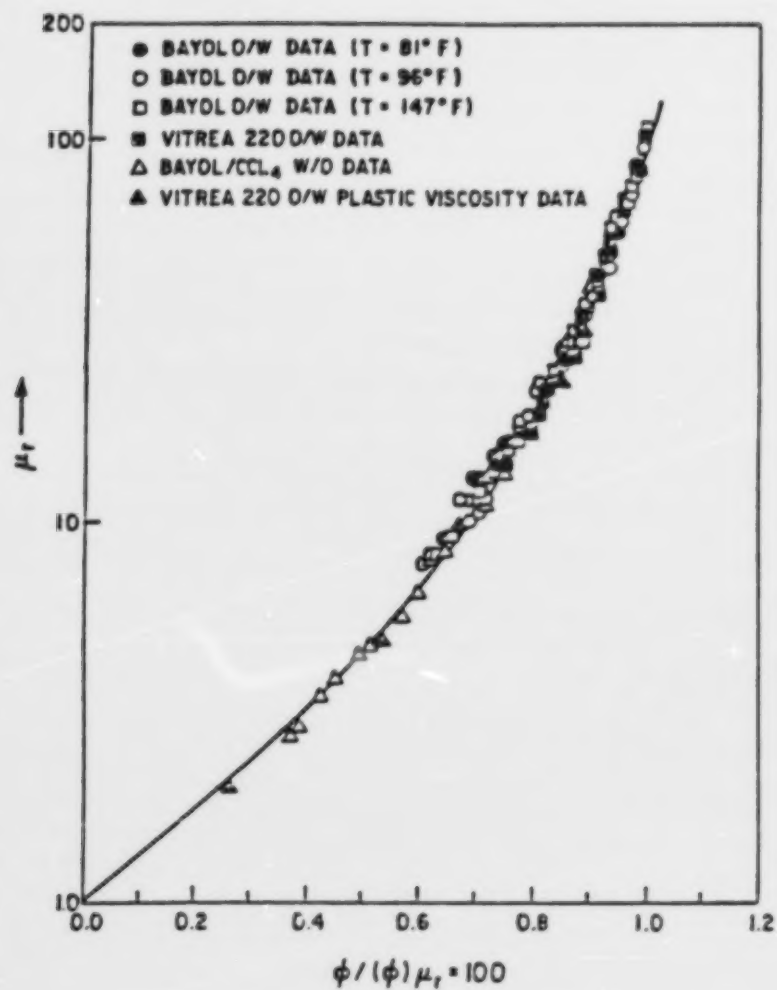


Figure 10: Relative viscosity versus the normalized concentration

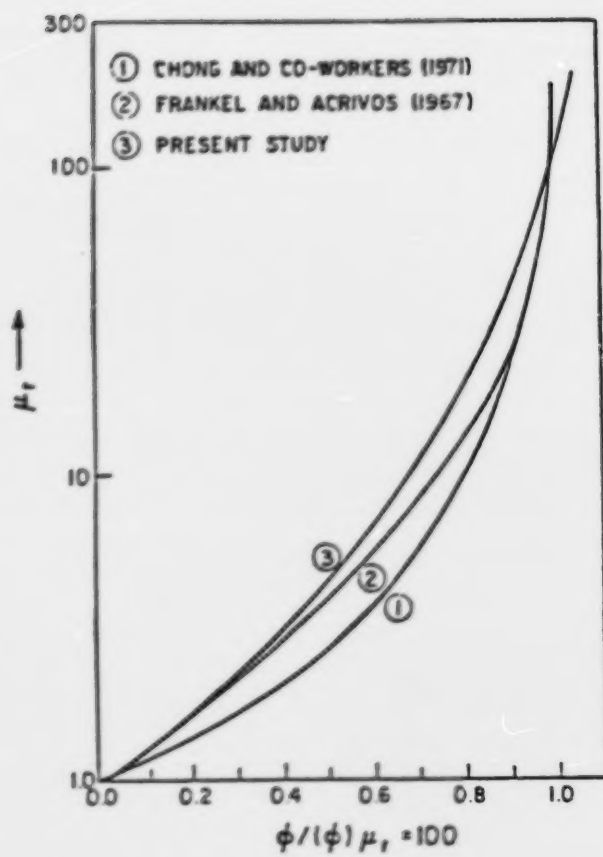


Figure 11: Comparison of suspension viscosity models with the present emulsion viscosity model

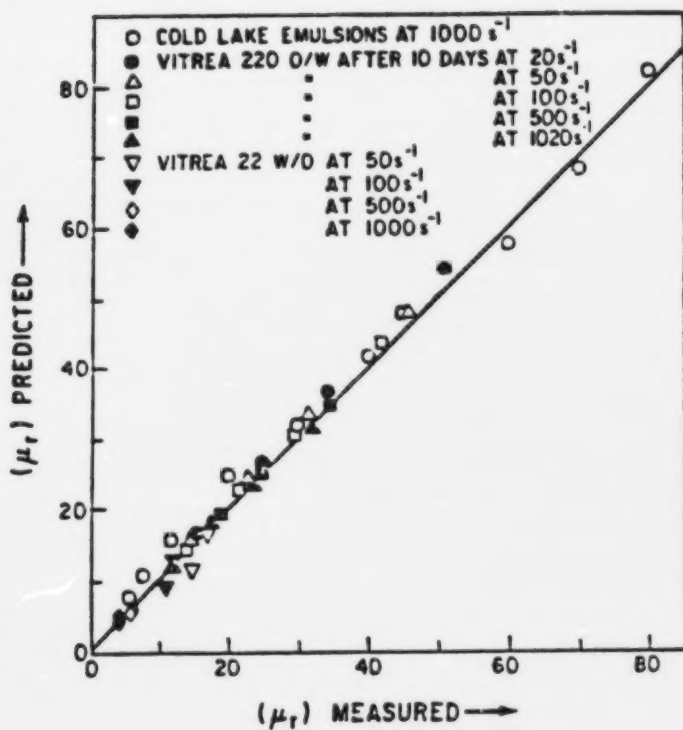
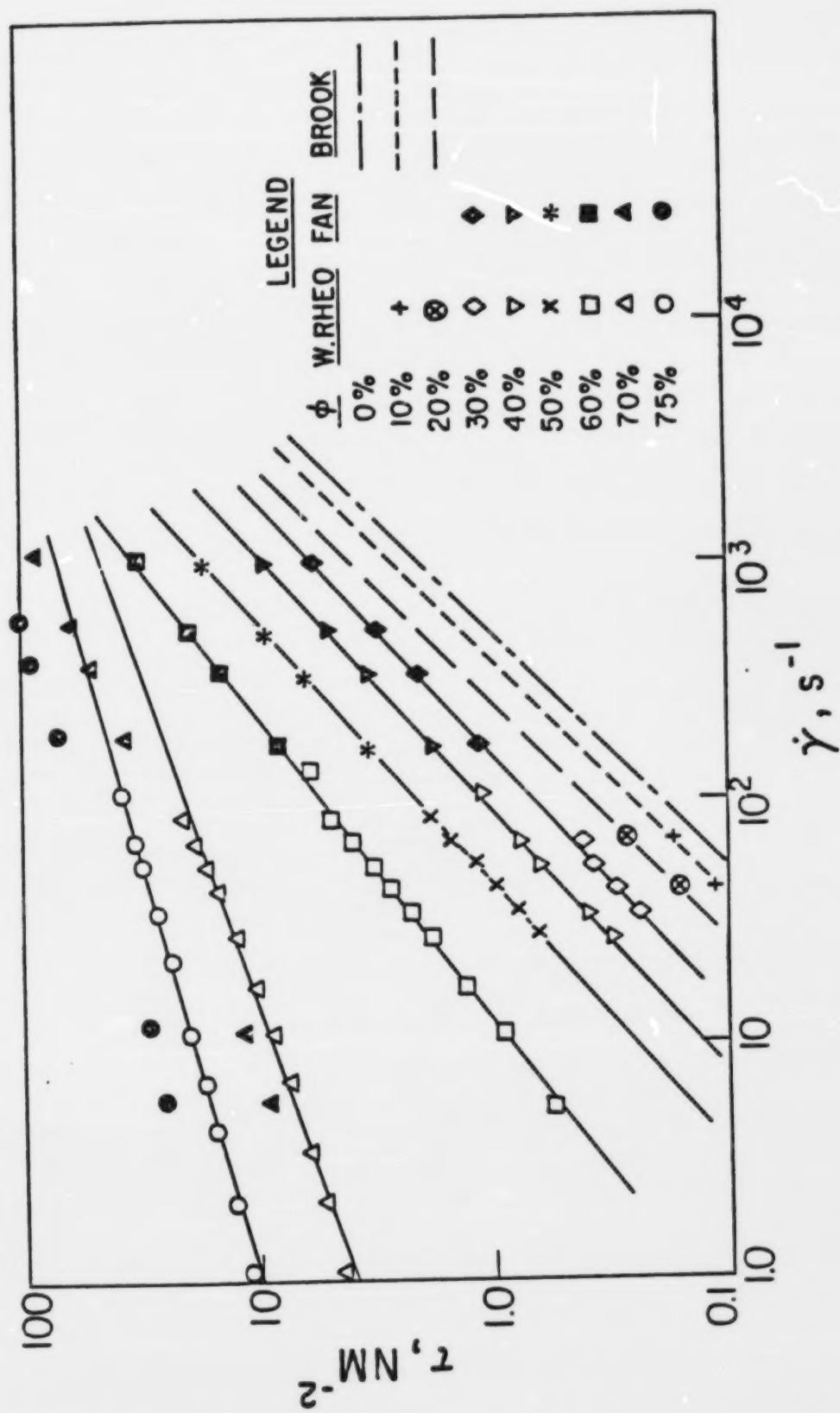
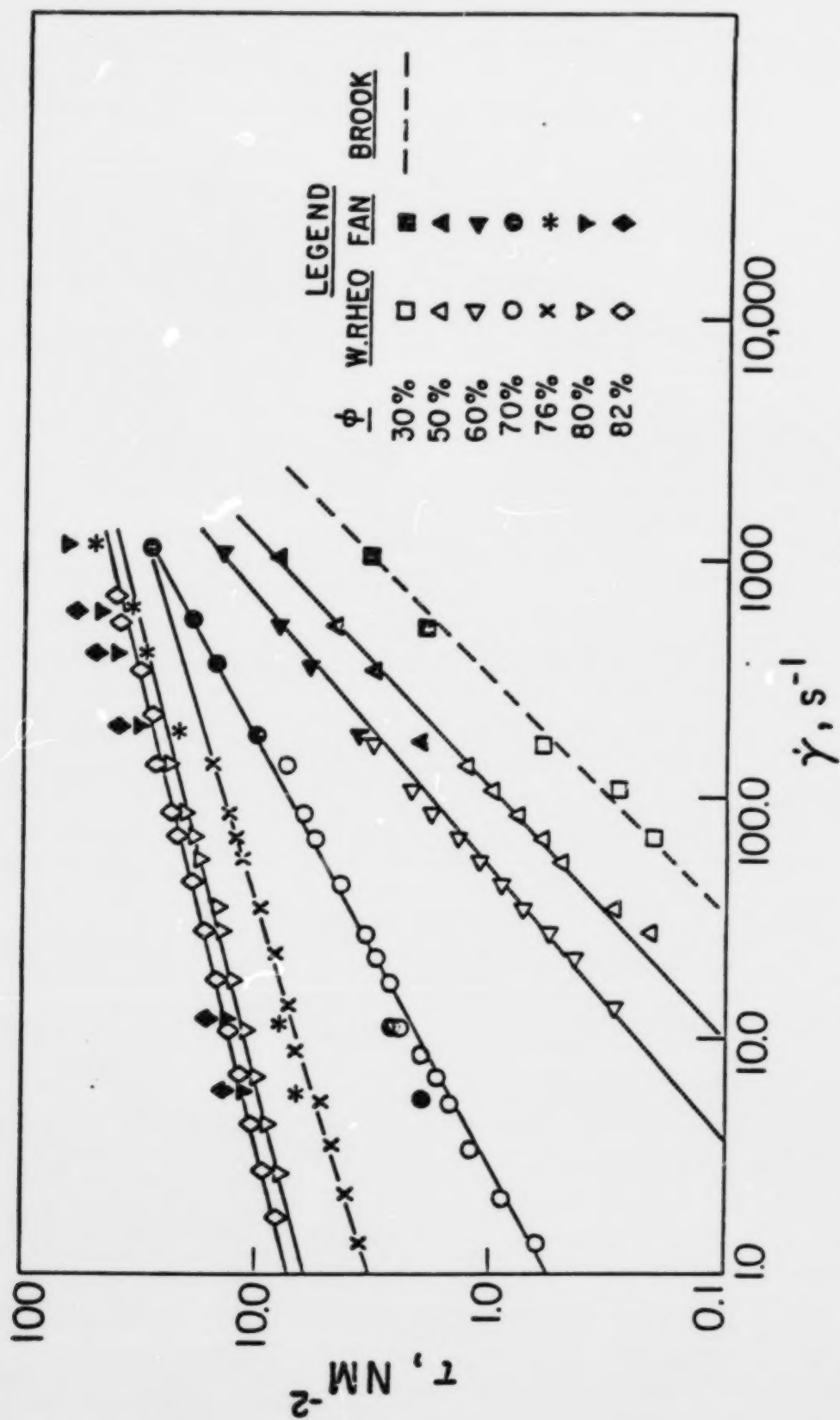
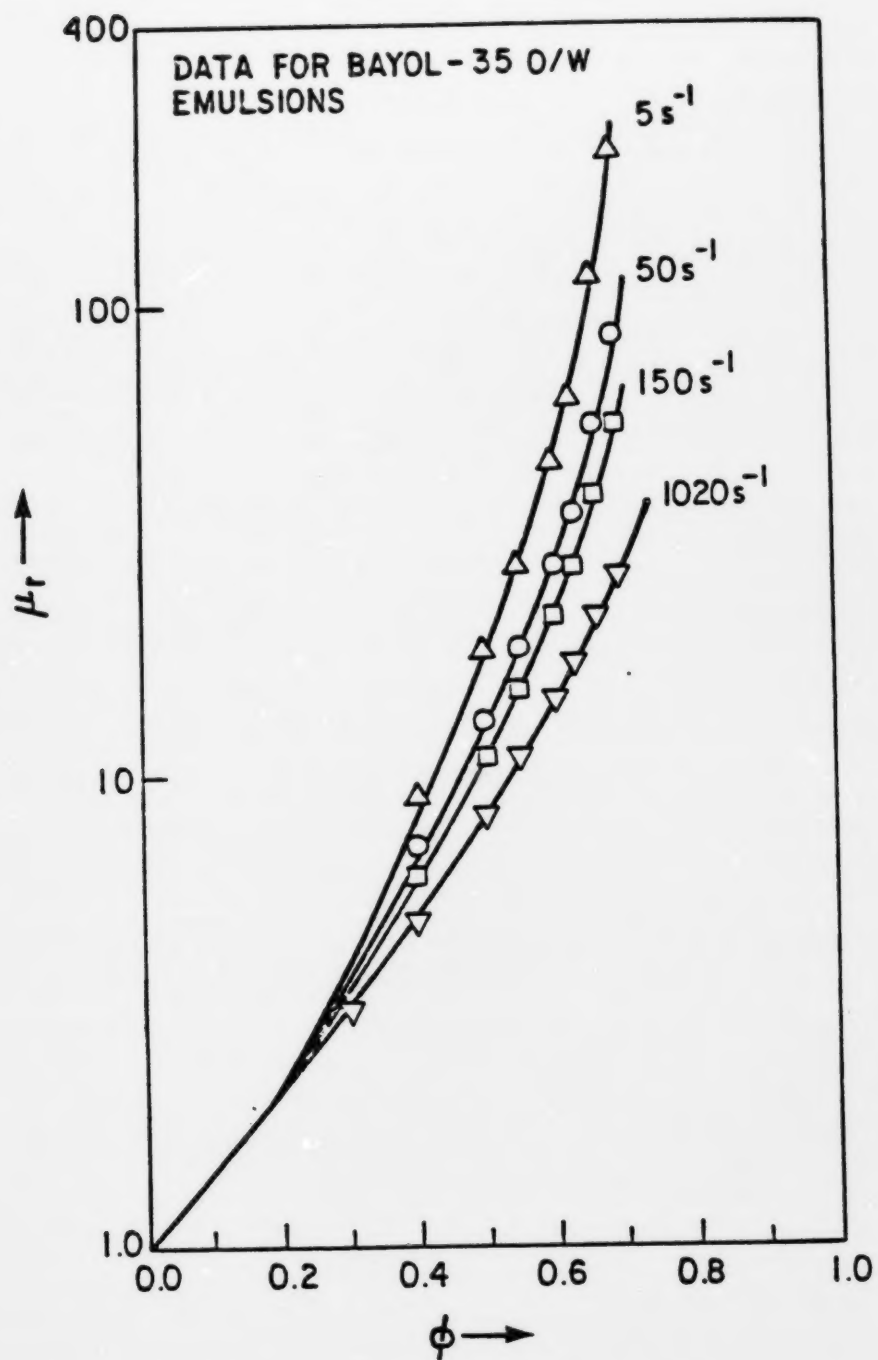
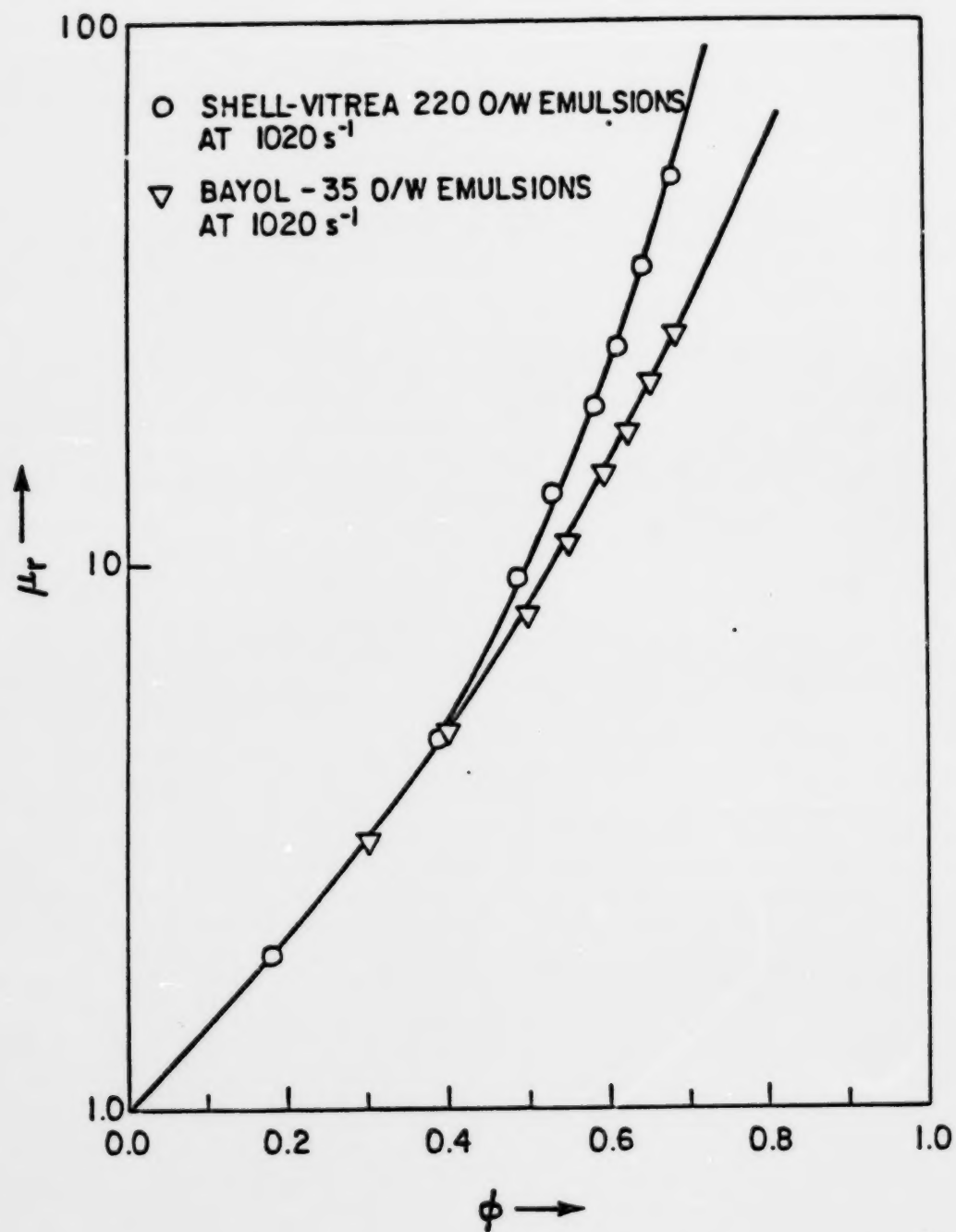


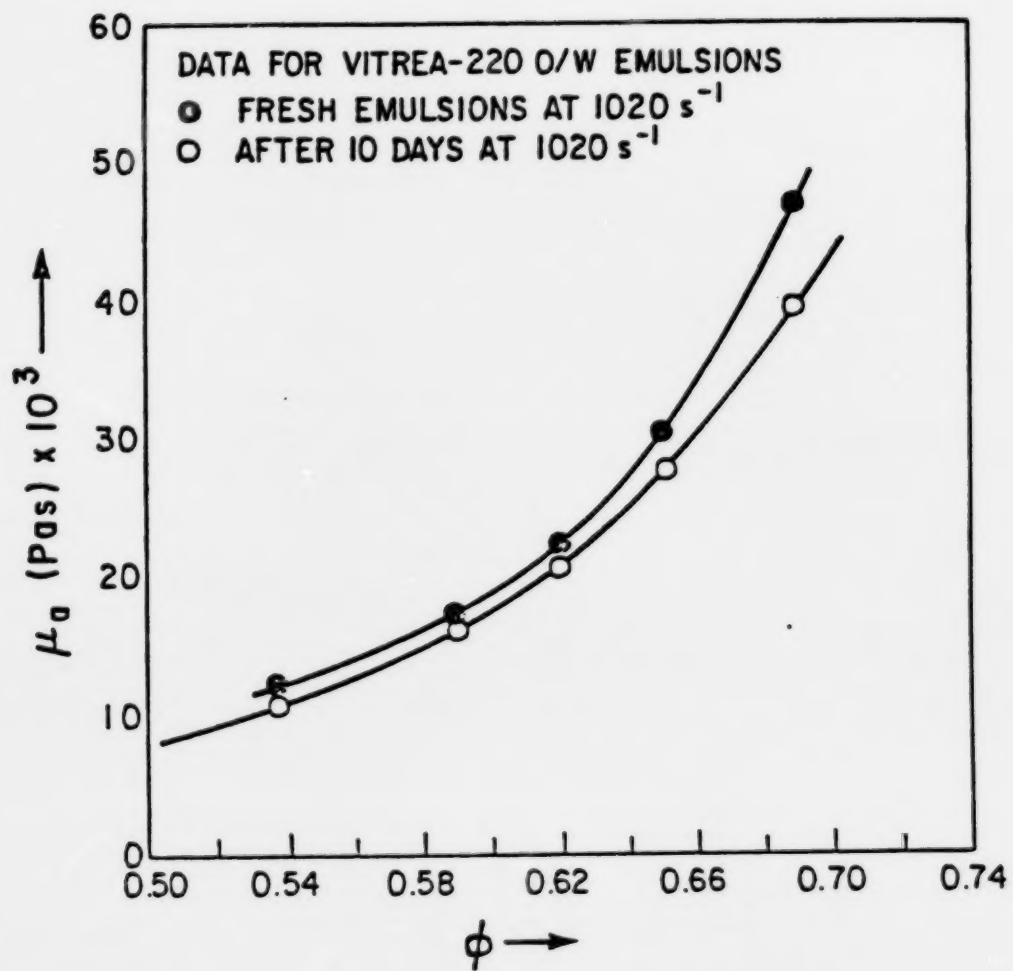
Figure 12: Predicted relative viscosity versus the measured relative viscosity

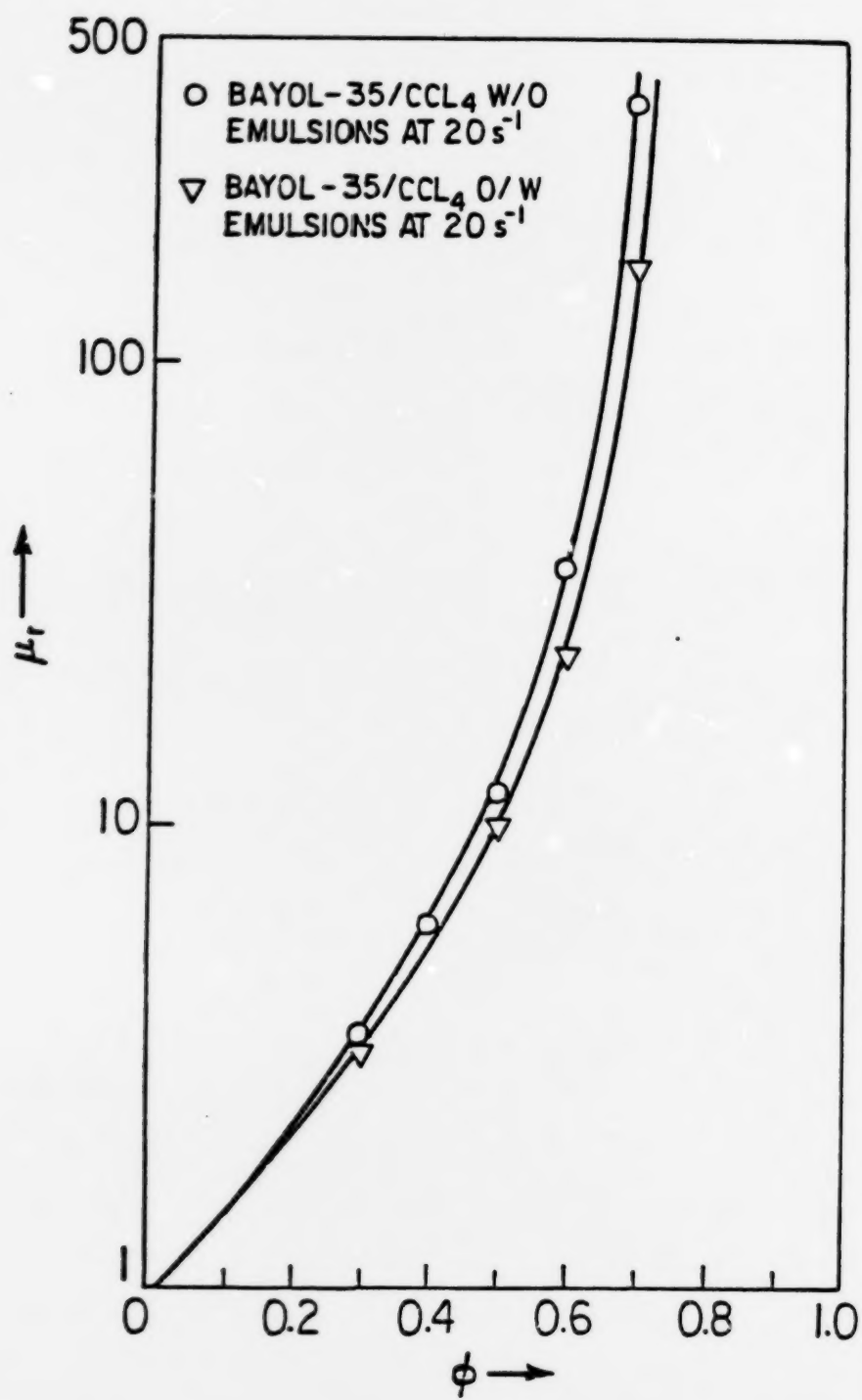


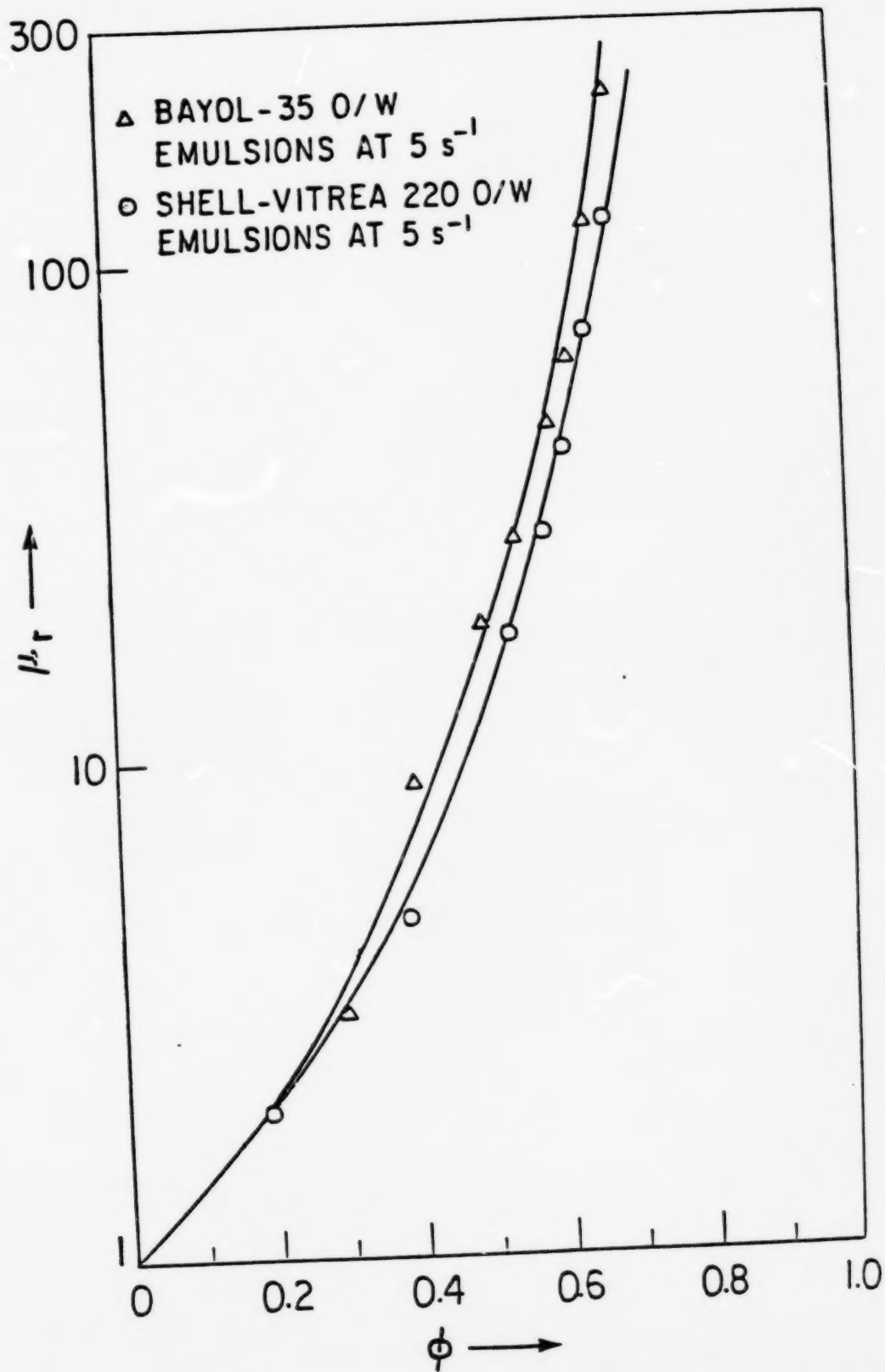


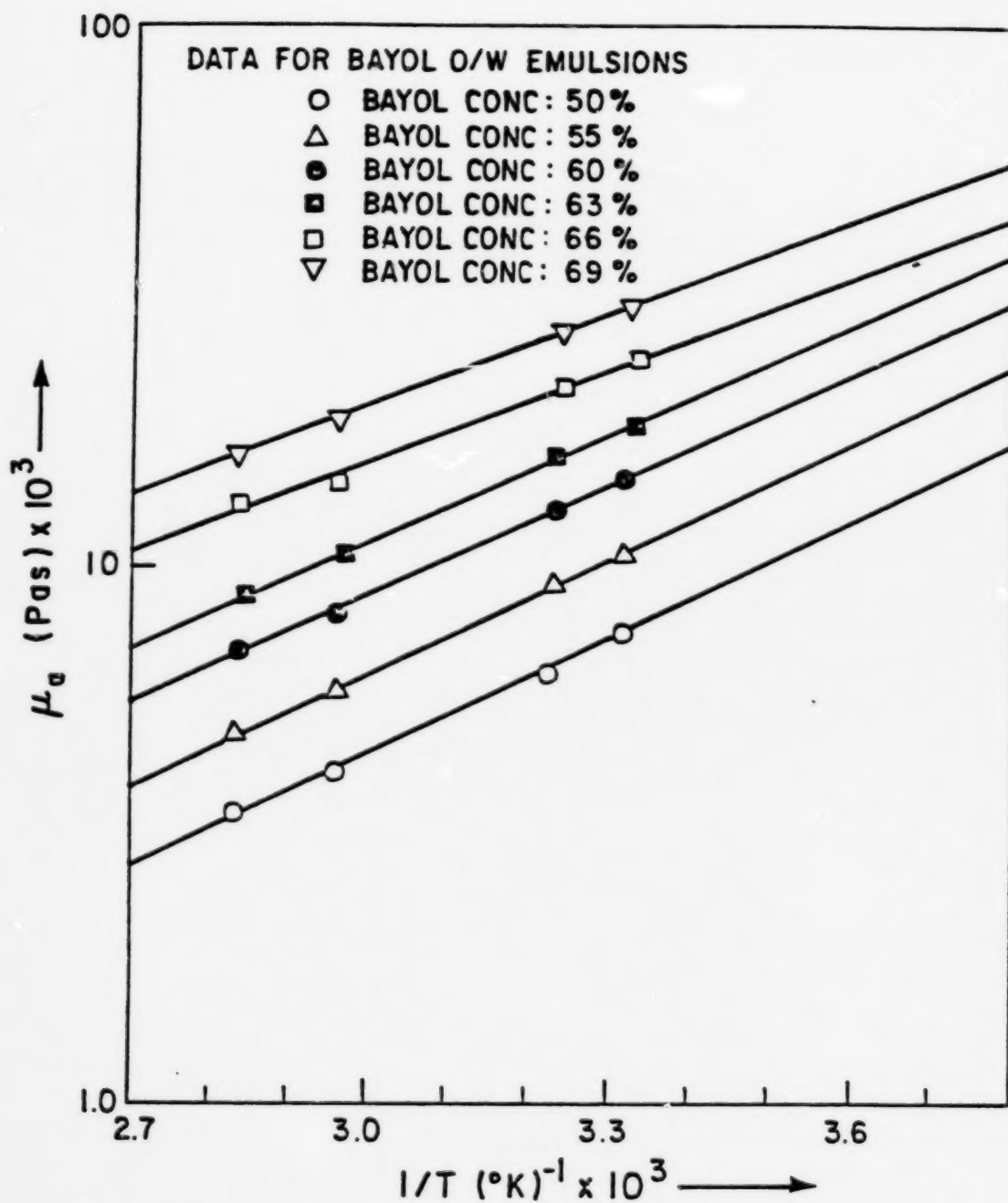


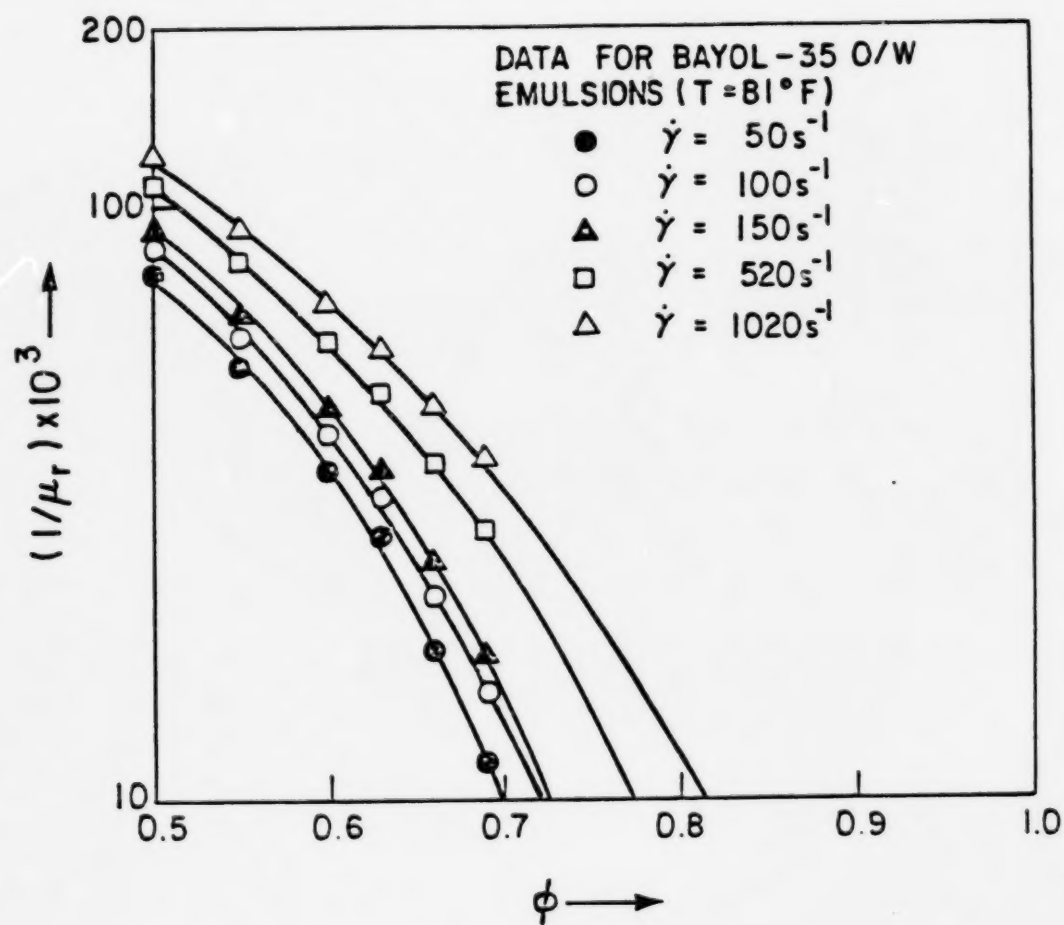


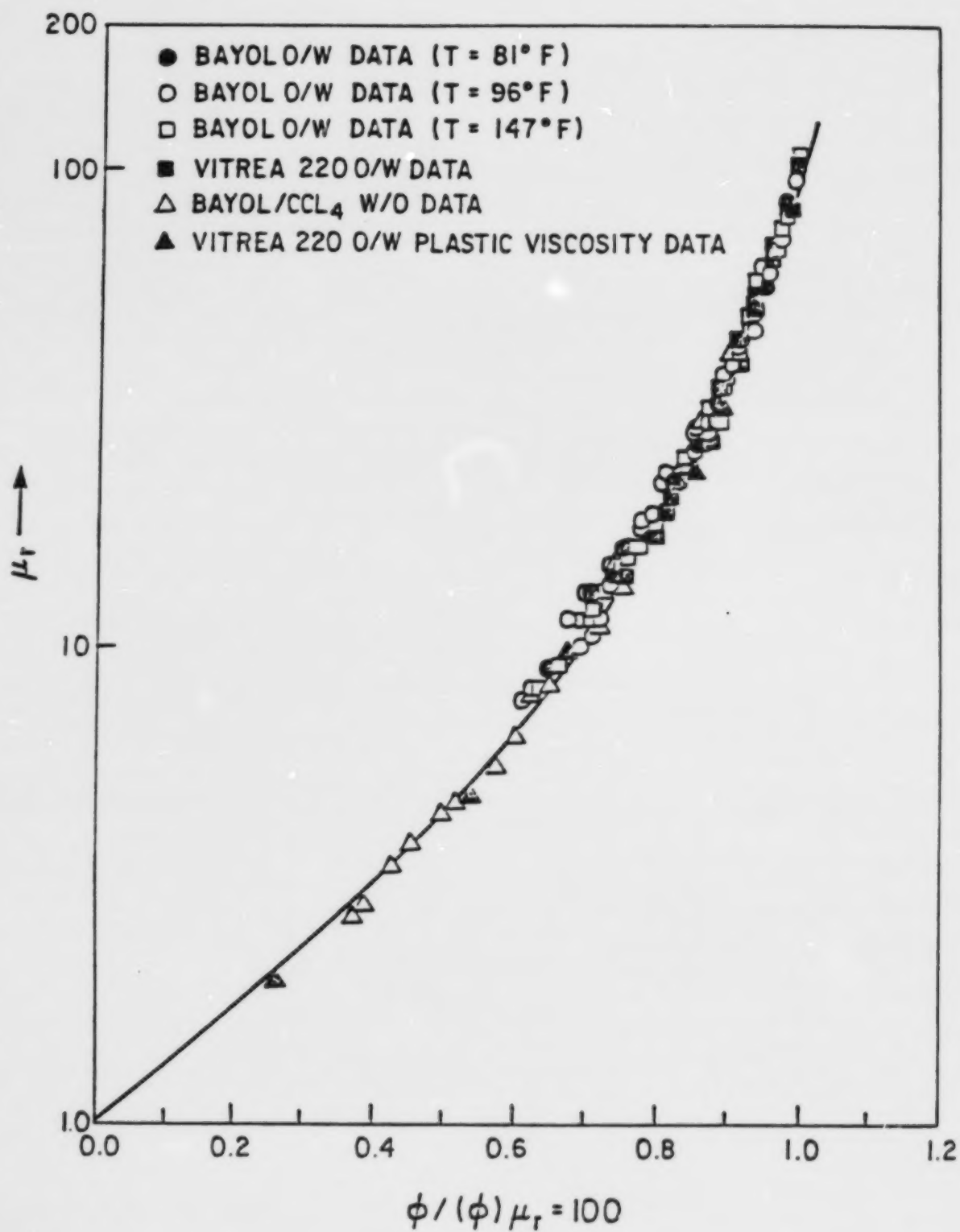


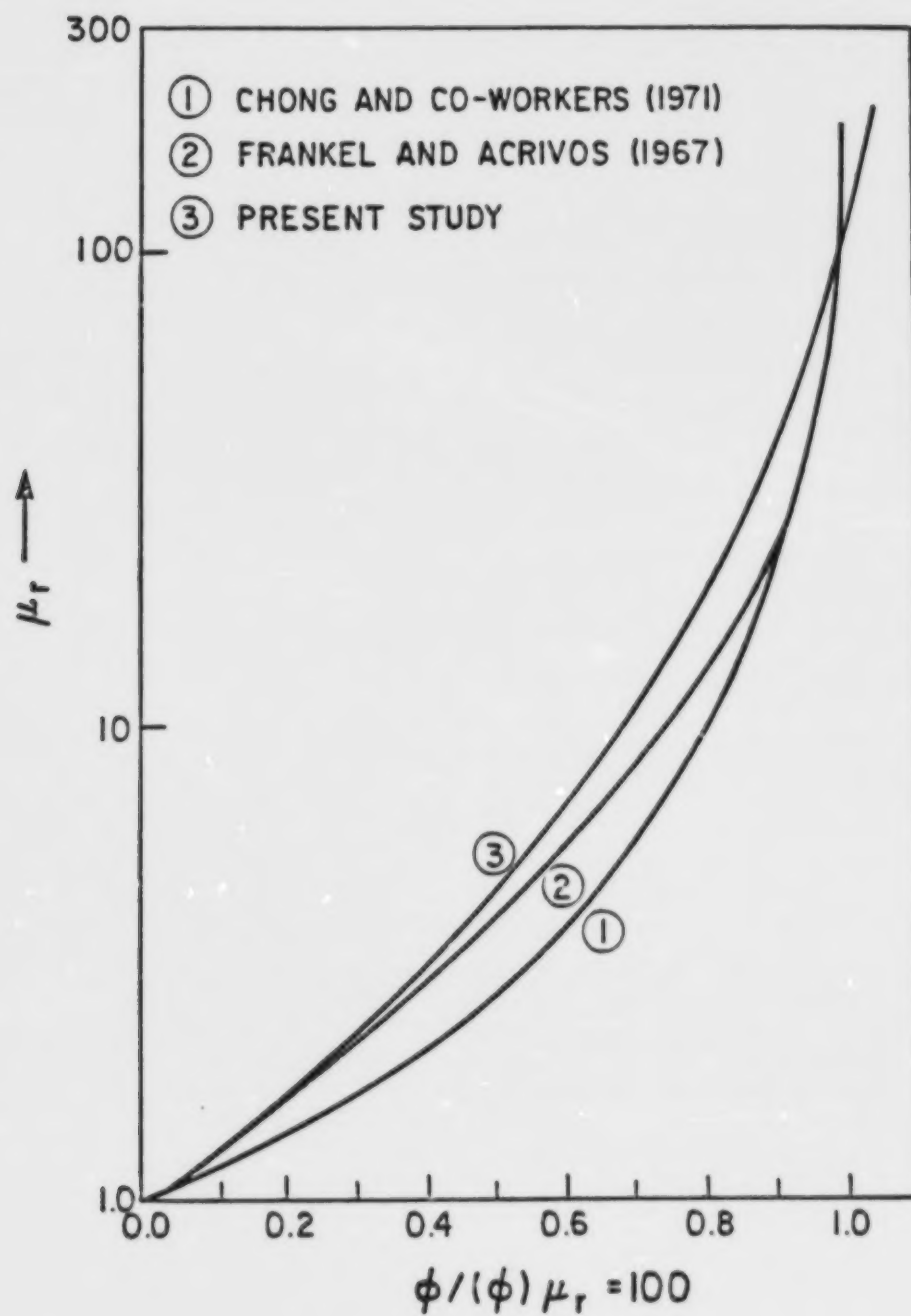


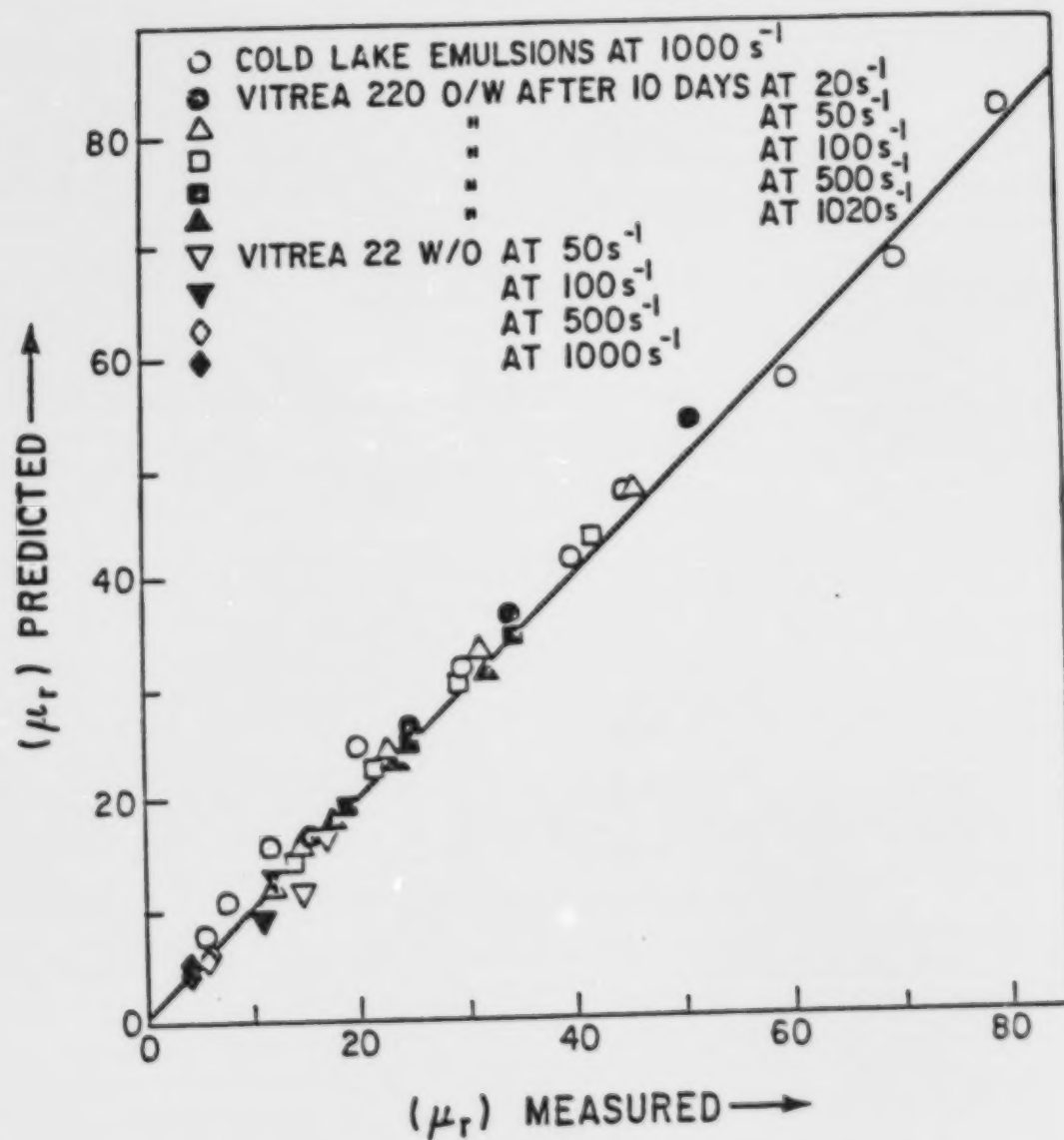












PAPER 7

ON THE RHEOLOGY OF HEAVY OIL AND ITS EMULSIONS

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ABSTRACT

The Rheological properties of a heavy oil and the heavy oil in tap water emulsions were studied using a coaxial cylinder viscometer. The variation of these properties with temperature and aging was investigated. The effects of oil concentration and added sodium chloride on the viscosity of the emulsions were also investigated.

The results show that heavy oil behaves like a Newtonian fluid and its viscosity is very sensitive to temperature. On aging, the viscosity of oil is found to increase significantly and it exhibits slight pseudoplastic behaviour.

The apparent viscosity of an emulsion decreases with the temperature. On aging, the viscosity of the emulsion first increases dramatically and then decreases with further aging time. Some thixotropic behaviour is also seen in these aged emulsions. The increase in oil concentration leads to an increase in the viscosity of the emulsion. Addition of sodium chloride to emulsions reduce their viscosity dramatically.

INTRODUCTION

Most of the crude oils (especially those recovered by secondary and tertiary oil recovery methods) are produced in the form of stable emulsions. In situ created emulsions are, in fact, considered as potential tools to the enhancement of the oil recovery process. The study of rheological properties of these emulsions is very important in order to predict their flow behaviour in pipelines and porous media.

The objectives of this study were:

1. To determine rheology of a heavy crude oil and to study the effects of temperature and aging on the viscosity of the oil.
2. To determine the rheology of heavy oil in tap water emulsions and study the effects of oil concentration, aging, sodium chloride and temperature on the rheological properties of the emulsions.

EXPERIMENTAL WORK

Oil

The oil used in this study was a Canadian heavy oil. The oil was recovered by the primary oil recovery method and was supplied to us by PETRO-CANADA in May, 1984. The oil had a density of 959 kg/m^3 and a viscosity of 1075 mPa.s at 25°C .

Emulsifier

The commercially available surfactant TRITON X-100 was used to stabilize the heavy oil-in-tap water emulsions. TRITON X-100 (iso-octylphenoxypolyethoxy ethanol) is a non-ionic water soluble emulsifier.

Emulsification Procedure

The emulsions were prepared by using the agent-in-water method. 1% by volume of Triton X-100 based on the water phase (tap water) was used to

stabilize the emulsions. Triton x-100 was dissolved in water, and to this solution, the required amount of heavy oil was added while the mixture was sheared in a high shear machine (1-LV Gifford-Wood homogenizer) for 15 minutes at a speed of about 3500 r.p.m. To obtain higher concentration emulsions, more oil was added to the existing lower concentration emulsion and the mixture sheared again for 15 minutes at a speed of 3500 r.p.m.

Viscosity Measurement

The rheology of heavy oil and heavy oil emulsions was determined by using a Fann coaxial cylinder viscometer. The model used in this study was 35A/SR12. This model had 12 test speeds ranging from 0.9 r.p.m. to 600 r.p.m. The outer cylinder (Rotor R-1) had a radius of 1.8415 cm and was made of stainless steel. The inner cylinder (Bob B-1) was also made of stainless steel and had a radius of 1.7245 cm. The height of the bob was 3.8 cm and the gap between the rotor and the bob was 1.17 mm. For this rotor and bob combination, the shear rates could be varied from 1.53 s^{-1} to 1022.04 s^{-1} . The instrument was also provided with a special electrically heated sample cup in order to study the effect of temperature on the rheology of fluids.

Density Measurement

The density of heavy oil was determined by using a Fisher pycnometer made of aluminium alloy. The volume of the pycnometer cavity was determined by calibrating it with water.

RESULTS AND DISCUSSION

Heavy Oil Rheology

Figure 1 shows the viscosity of the heavy oil as a function of temperature. It is obvious from the figure that the viscosity of the crude

oil is very sensitive to temperature. The viscosity is reduced by 50% by heating the oil by about 10°C.

In order to study the effect of aging on the viscosity of the oil, the oil was first heated to about 83°C and was then left to cool down to room temperature overnight. On the next day the viscosity data was collected for this aged oil at room temperature. Figure 2 shows the comparison of viscosity data of both the fresh heavy oil (i.e. unheated oil) and the aged oil. The lower viscosity line is for the fresh oil (unheated) at room temperature (24°C). Increasing the shear rate has practically no effect on the viscosity of fresh oil indicating that the oil behaves like a Newtonian fluid at room temperature. The higher viscosity line is for the aged oil at room temperature. Upon aging (first heating the oil to a high temperature and then allowing it to cool down) the viscosity of the oil has increased by 22%. The aged oil is also seen to exhibit slight pseudoplastic behaviour. It should be noted that when the unheated oil (i.e. fresh oil) was left at room temperature overnight, the viscosity was practically unaffected.

The possible reason for the increase in viscosity upon heating/aging is the oxidation of heavy oil. A similar type of behaviour was observed by Steinborn and Flock (1) for the Epping crude oil. Maschopedis and Speight (2) also observed that the viscosity of heavy oil was significantly increased when it was exposed to oxygen at high temperature. They found that oxygen acts primarily as a catalyst in the production of asphaltenes. It is now a well known fact (3) that as the high molecular weight asphaltene fraction in the oil increases, the viscosity increases. Also, it is this asphaltene fraction which leads to non-Newtonian behaviour of the oil. The effect of oxidation on the viscosity of heavy oil is also

described in a very recent article by Rambabu and Cormack (4). They found that even at a relatively low extent of oxidation, the viscosity of heavy oil could be more than two orders of magnitude higher than that of an unoxidized one.

Oil-in-Water Emulsion Rheology

Figure 3 shows the variation of the apparent viscosity of fresh emulsions (data was collected immediately after preparation of the respective emulsion) as a function of shear rate.

Emulsions with dispersed phase volume concentrations (i.e. oil concentrations) up to 56% were found to be Newtonian in the range of shear rates studied (100 s^{-1} - 1022 s^{-1}). It is necessary to point out that these emulsions may behave like non-Newtonian fluids at shear rates lower than 100 s^{-1} and most of the structure breakdown (i.e. deflocculation of floccs of dispersed particles) may occur in that low shear rate range. However, because of the low torque developed at low shear rates we could not collect any data in that region. For oil concentrations greater than 56% by volume, the emulsions behaved like pseudoplastic non-Newtonian fluids and the viscosities decreased with increasing the shear rate.

Figure 4 shows the variation of apparent viscosity of fresh emulsions with the oil phase concentration at a shear rate of 1022.04 s^{-1} . It is obvious from the figure that the viscosity of the emulsion increases with the oil concentration and the rate of change of viscosity with concentration increases as the oil concentration increases.

Effect of Aging On the Viscosity of Emulsions

Figure 5 shows a plot of the apparent viscosity of a 70% emulsion as a function of time. The viscosity of the emulsion is first found to increase

as the aging time increases and then it starts decreasing with further aging. Aged emulsion also exhibits some thixotropic behaviour i.e. at any aging time, the viscosity is a function of shear rate as well as the duration of shear. The scatter in viscosity data (Figure 5) for a particular aging time and a particular shear rate is due to this thixotropic behaviour.

To make sure that the increase in viscosity of the emulsion was not because of an inversion of oil-in-water emulsion to a water-in-oil type during the aging process, a dilution test was performed. The emulsion was found to be readily dispersible in the water phase indicating that the continuous phase was water and therefore the emulsion was of the oil-in-water type. The emulsion was found to be indispersible in the oil phase, again indicating that the continuous phase was water. It can, therefore, be concluded from these experiments that there was some permanent structure formation in the emulsion with aging. Probably, the degree of flocculation was increased with aging time and this led to higher viscosities. Since we used tap water in the preparation of the emulsions, the dissolved minerals and ions could be responsible for the aging effects observed. However, the exact mechanism on how these dissolved materials and ions lead to structure formation is not known at this time.

It seems from Figure 5 that the structure formation process continues for about 35 hours while the viscosity of the emulsions continues to rise. However, after this time, the viscosity starts decreasing with further aging time. The decrease in viscosity is probably because of an increase in the average particle size of the emulsion.

Figure 6 shows the aging effect on the viscosity of 63.5% emulsion. The figure shows that the viscosity became almost twice the value of fresh

emulsion within 6 1/2 hours. Further aging time increased the viscosity further. The degree of non-Newtonian behaviour (i.e. pseudoplastic nature) also increased with the aging time indicating that some permanent structure formation (i.e. increased flocculation) definitely occurred with aging.

Effect of Salt on the Viscosity of Emulsions

In figure 7 is shown the viscosity data of aged 70% emulsion ($t = 72.5$ hours) with no NaCl addition and with NaCl addition. Two different concentrations of NaCl were used viz: 1.55% by weight and 2.75% by weight based on the total weight of the emulsion. For aged emulsion with no NaCl addition, two curves are shown. The one with the higher viscosity was obtained without preshearing (i.e. the data was collected immediately) and the one with the lower viscosity was obtained with preshearing at 511.02 s^{-1} for about 10 minutes (further shearing did not decrease the viscosity). It is clear from these two curves that there is some thixotropy in this emulsion and the viscosity depends upon the duration of shear as well as the shear rate.

On addition of NaCl to the emulsion, the viscosity was found to decrease remarkably. The middle two curves shown in Figure 7 were obtained when the NaCl concentration was 1.55% by weight based on the total weight of the emulsion. Here again the emulsion exhibited some thixotropy and therefore two curves were obtained depending upon whether the emulsion was presheared or not. If the emulsion was sheared at 511.02 s^{-1} for about 10 minutes (lower viscosity curve out of the two curves under discussion) the viscosities were lower. When the data was collected immediately without preshearing the viscosities were higher. The viscosity of the emulsion with NaCl is about one-half of the original value (i.e. of emulsion without NaCl).

Further addition of NaCl to this emulsion lowered the viscosity further. The lowest curve shown in the Figure 7 was obtained for the emulsion with a 2.75% by weight NaCl concentration. The data for this curve was obtained without preshearing. Since the emulsion was found to be unstable at high shear rates ($>350 \text{ s}^{-1}$), we could not collect any data. Figure 7 shows the effect of NaCl addition on the viscosity of aged 63.5% emulsion. Here again, the effect of salt was to decrease the viscosity.

It seems from these studies that the effect of NaCl addition is to break the structure existing in the emulsion. Probably, the degree of flocculation is greatly reduced on the addition of salt and the average particle size in the emulsion is also increased. Both of these factors will lead to a decrease in the viscosity of emulsion. The exact mechanism on how salt decreases the degree of flocculation or increases the average particle size is not known at this time. However, it is interesting to point out that our study with mineral oil in water emulsions (stabilized with Triton x-100) shows that the viscosity of mineral oil emulsions is practically unaffected by the addition of salt. From this it can be concluded that salt interacts with the heavy crude oil in some unique way that leads to structure breakdown in the emulsion. Further experiments are underway to determine the exact reasons.

Effect of Temperature on the Viscosity of Emulsions

Figure 9 shows the effect of temperature on the steady state viscosity of 70% aged emulsion. As the temperature is increased, the apparent viscosity was reduced. The decrease in the viscosity of emulsion with temperature was probably due to the following reasons.

1. the viscosity of the continuous phase decreases with temperature
2. the viscosity of the dispersed phase decreases with temperature

3. the average particle size of the emulsion may also increase upon heating

CONCLUSIONS

Based on the experimental data, the following conclusions are made:

1. The heavy oil studied behaves like a Newtonian fluid. On aging (first heating the oil and then allowing it to cool), the viscosity of the oil increases significantly and it then exhibits slight pseudoplastic behaviour.
2. The viscosity of an emulsion increases with oil concentration. The rate of increase of viscosity with oil concentration increases as the oil concentration increases.
3. On aging, the emulsions exhibit thixotropic behaviour. The steady state viscosity of an emulsion first increases with aging time and then starts decreasing with further aging time.
4. The viscosity of emulsions is considerably reduced by the addition of sodium chloride.
5. The effect of temperature is to reduce the viscosity of emulsions.

ACKNOWLEDGEMENTS

Financial support from CANMET is gratefully acknowledged by the authors. The authors also gratefully acknowledge Petro-Canada Resources for supplying a sample of heavy oil.

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4. Rambabu, D. and Cormack, D.E., "Effect of Oxidation on the Viscosity of Athabasca Bitumen", Can. J. Chem. Eng., Vol. 62, 562-564 (1984).

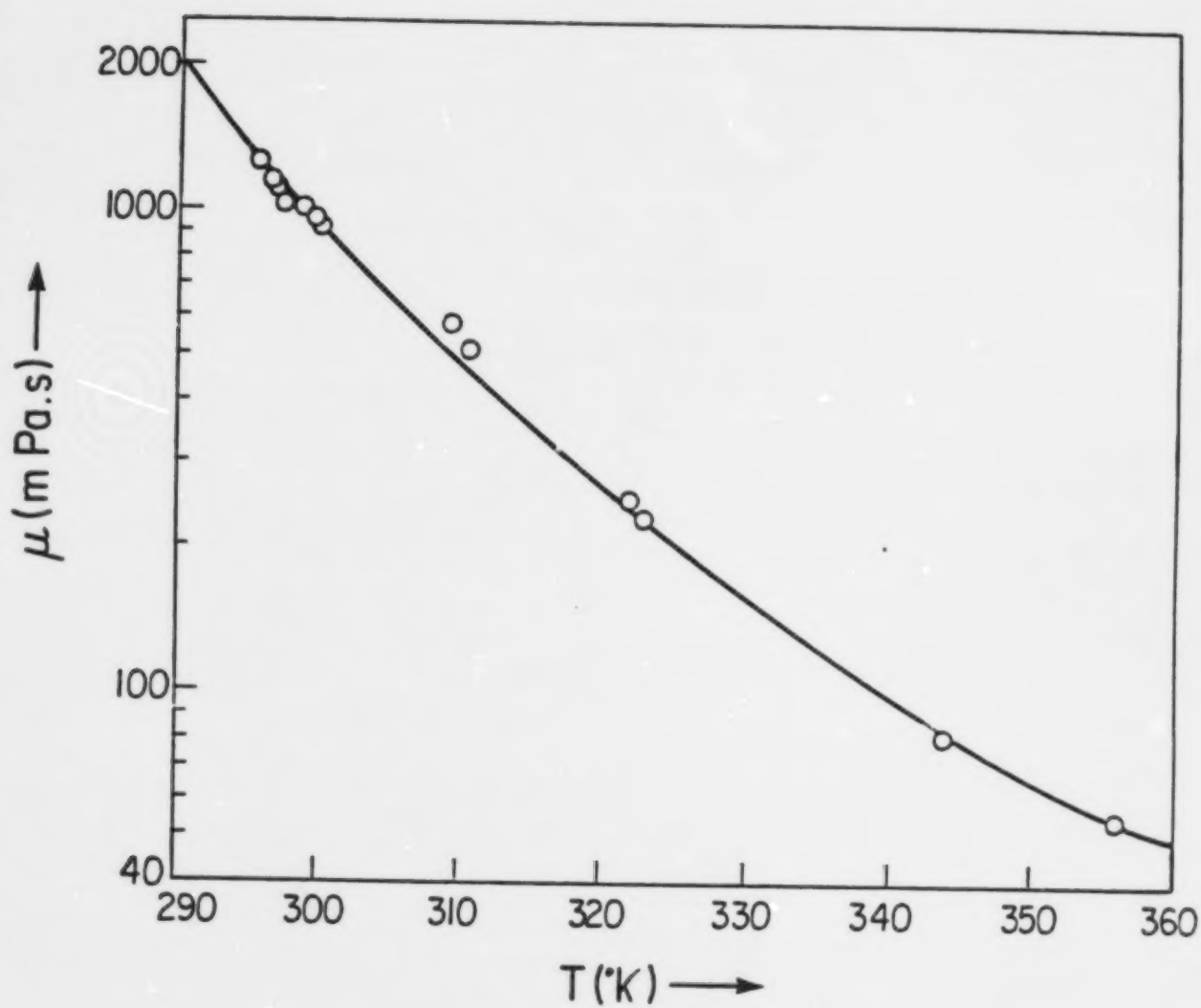


Figure 1. Viscosity of Bituminous Oil versus Temperature.

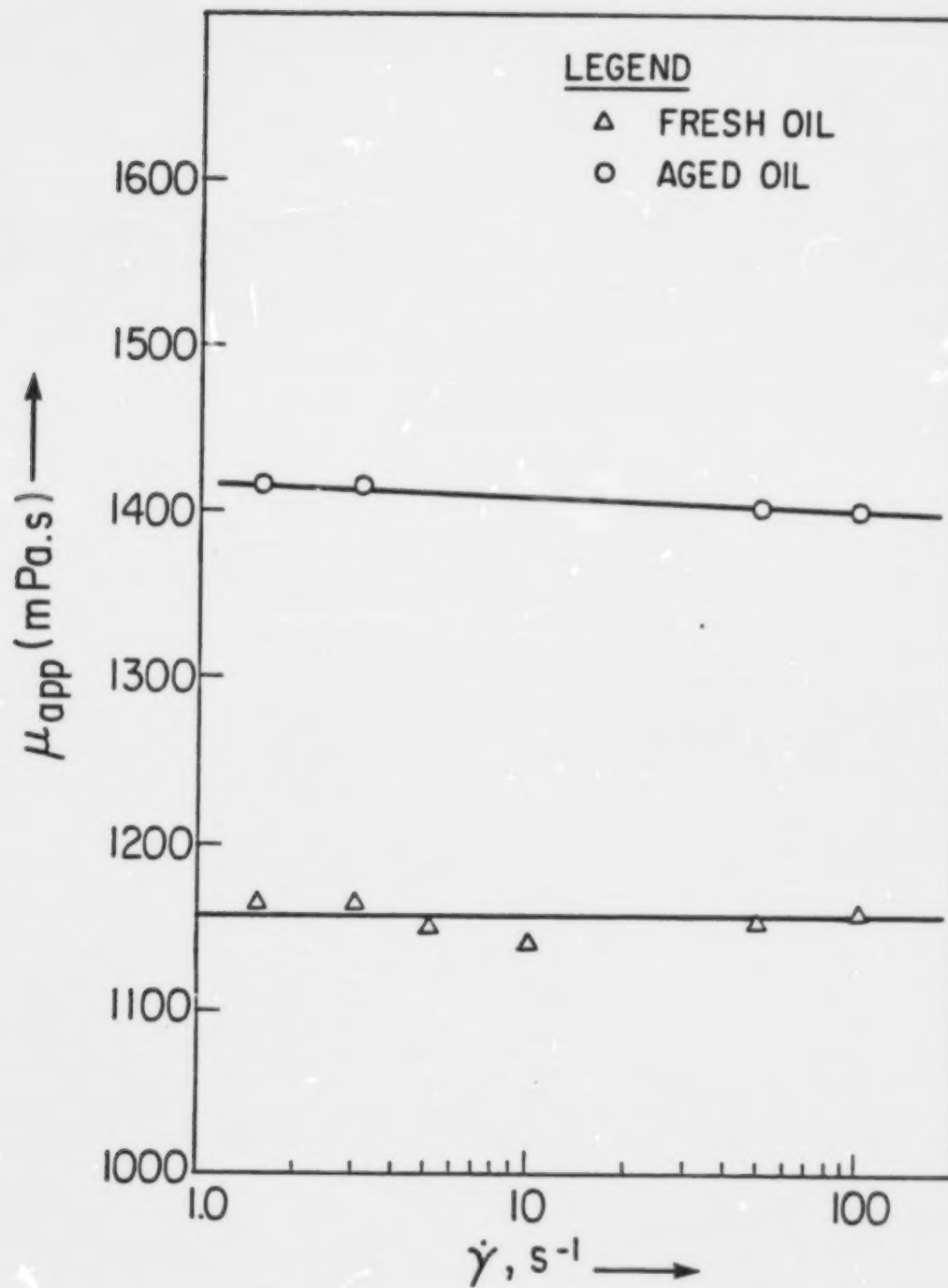


Figure 2. Apparent viscosity of oil before and after aging.

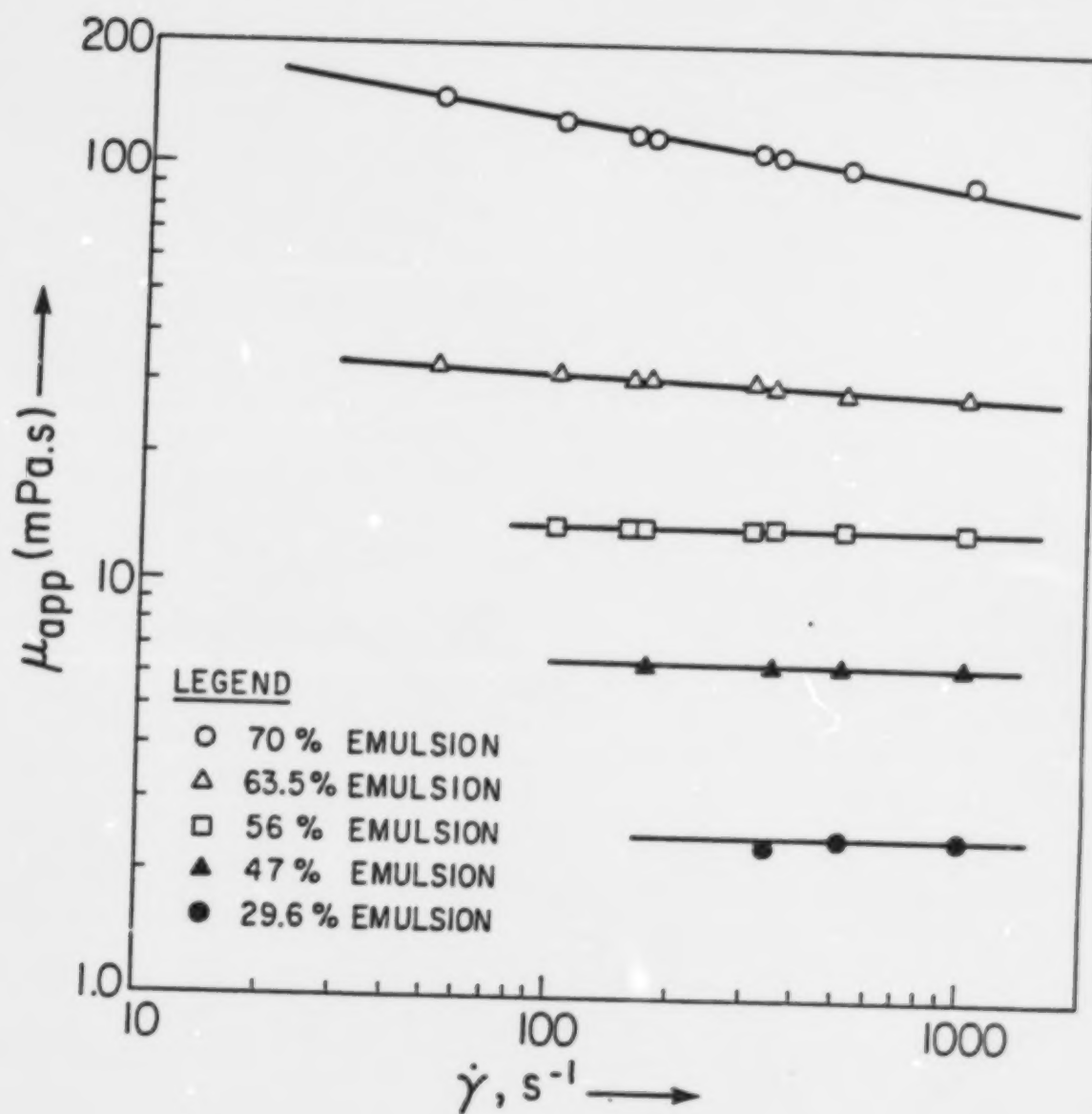


Figure 3. Apparent viscosity of emulsions of bituminous oil.

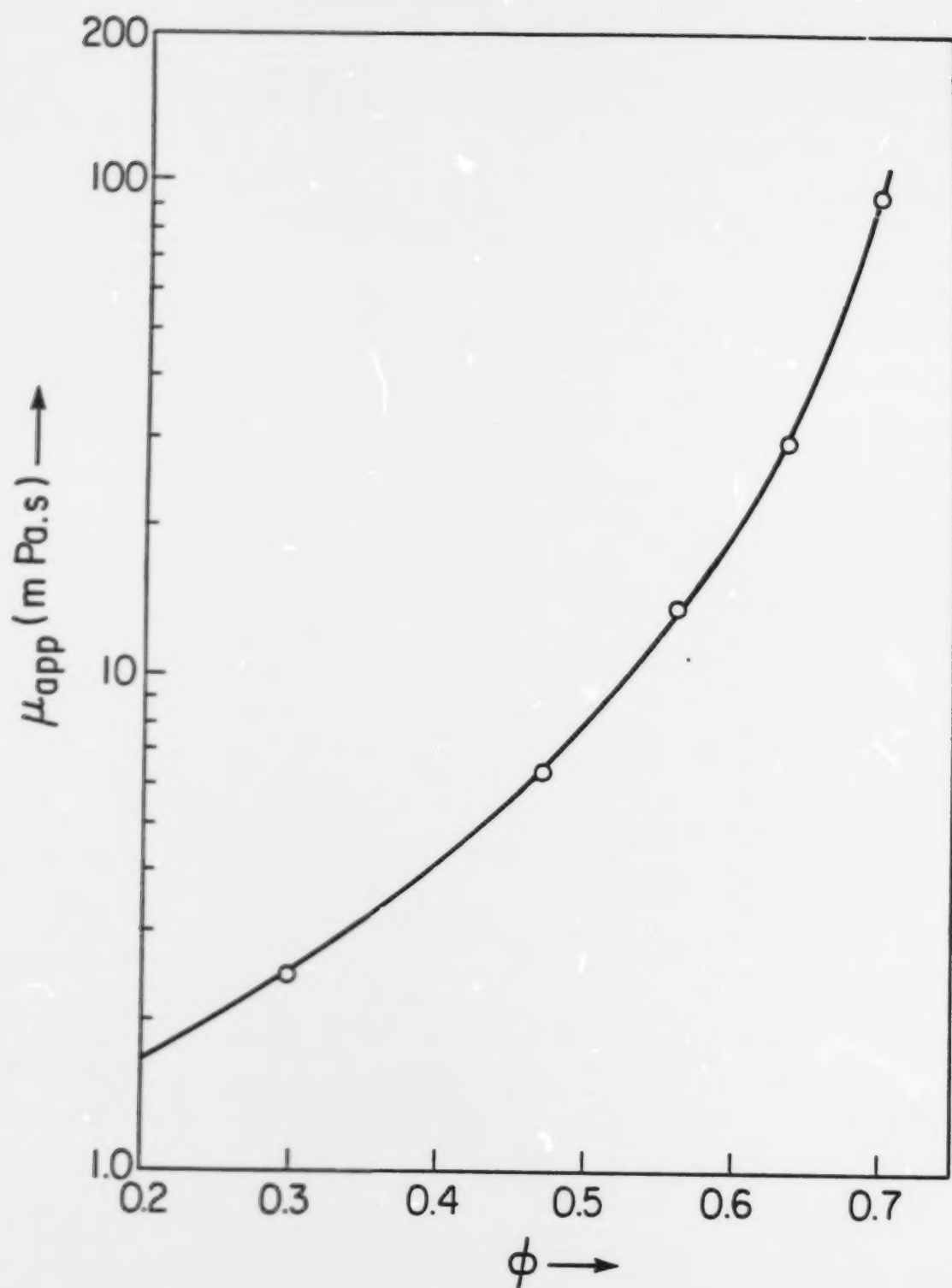


Figure 4. Apparent viscosity versus oil concentration.

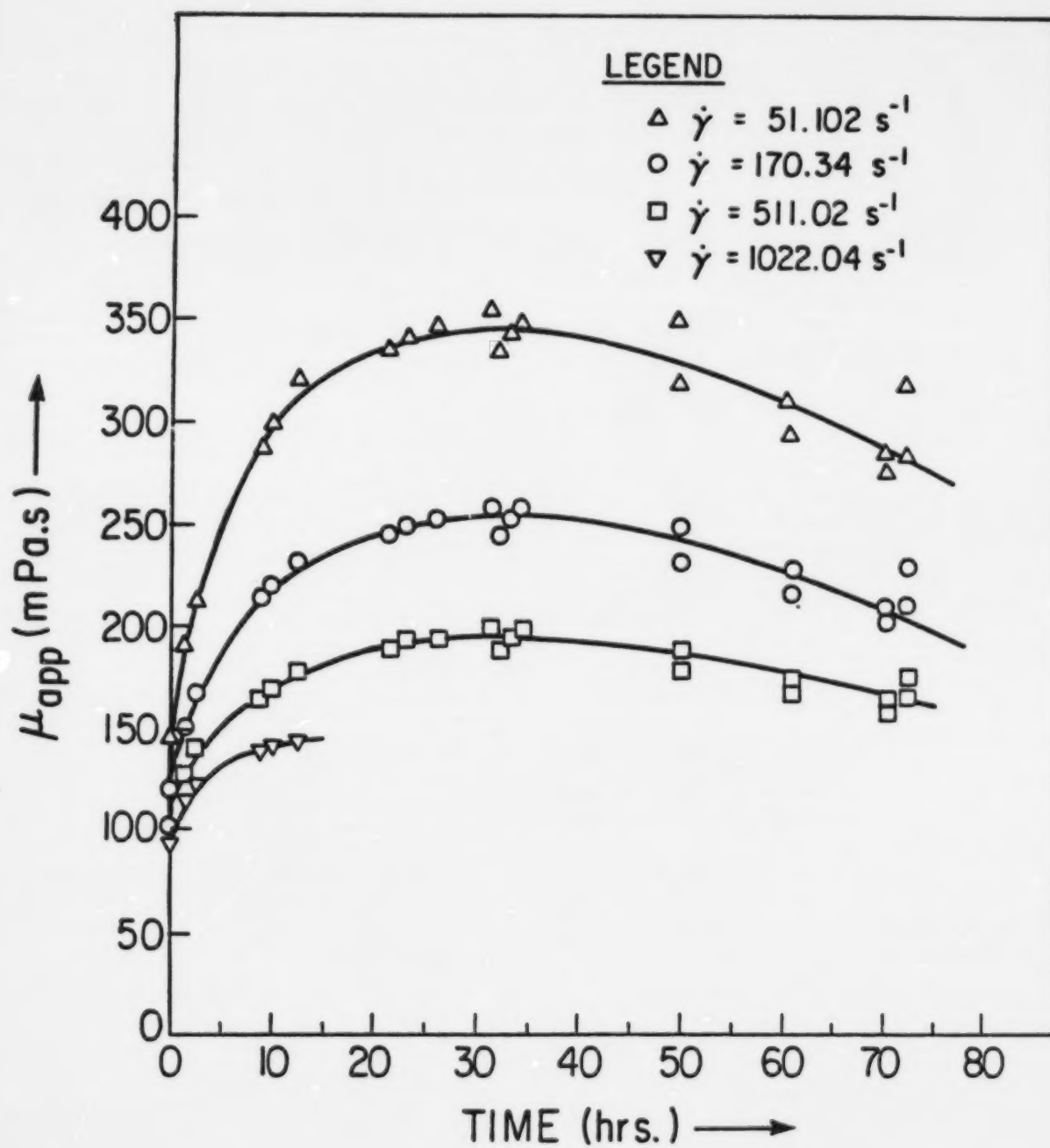


Figure 5. Effect of aging on emulsions at different shear rates.

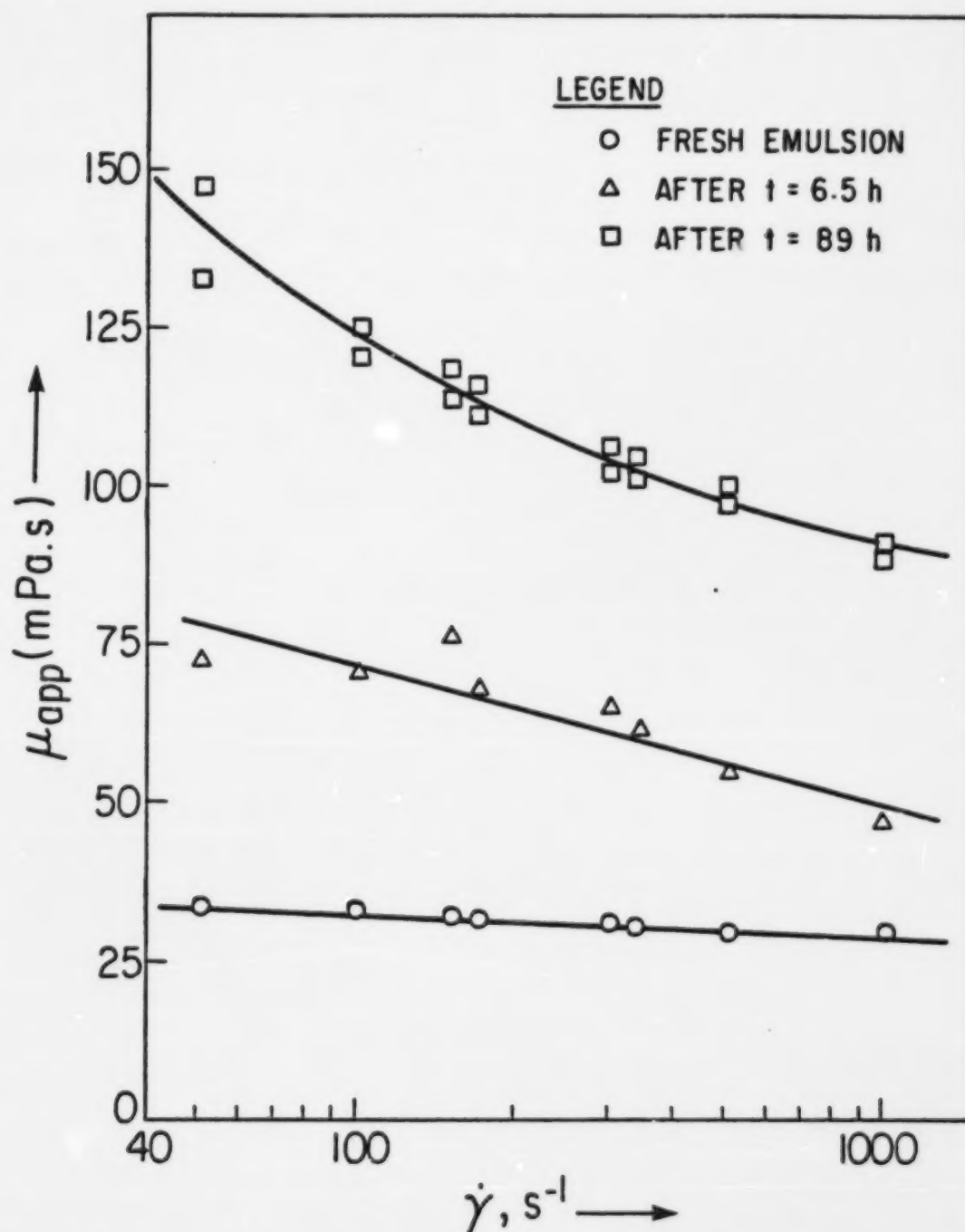


Figure 6. Flow curves of fresh and aged emulsions.

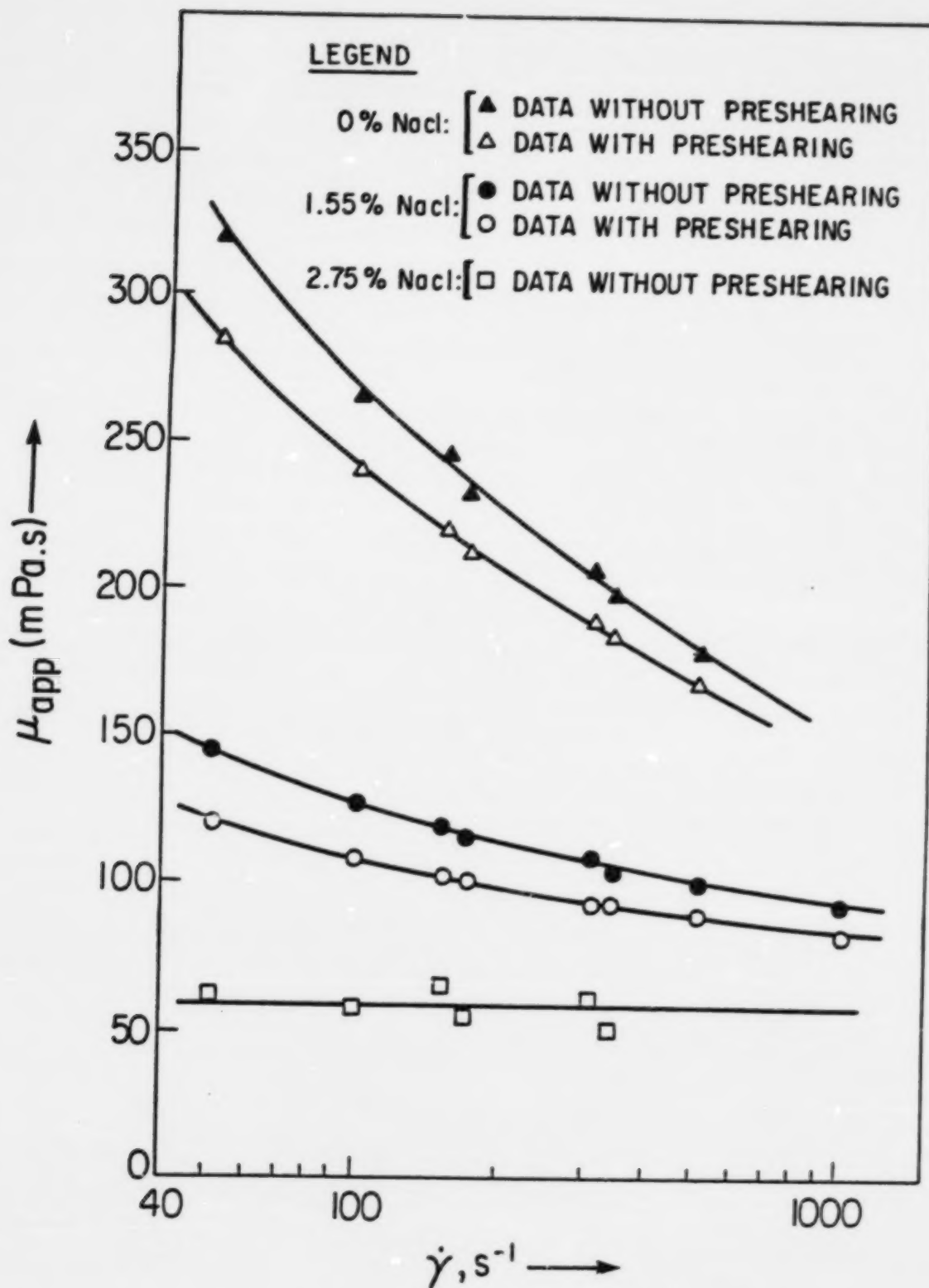


Figure 7. Effect of salts on sheared and unsheared emulsions.

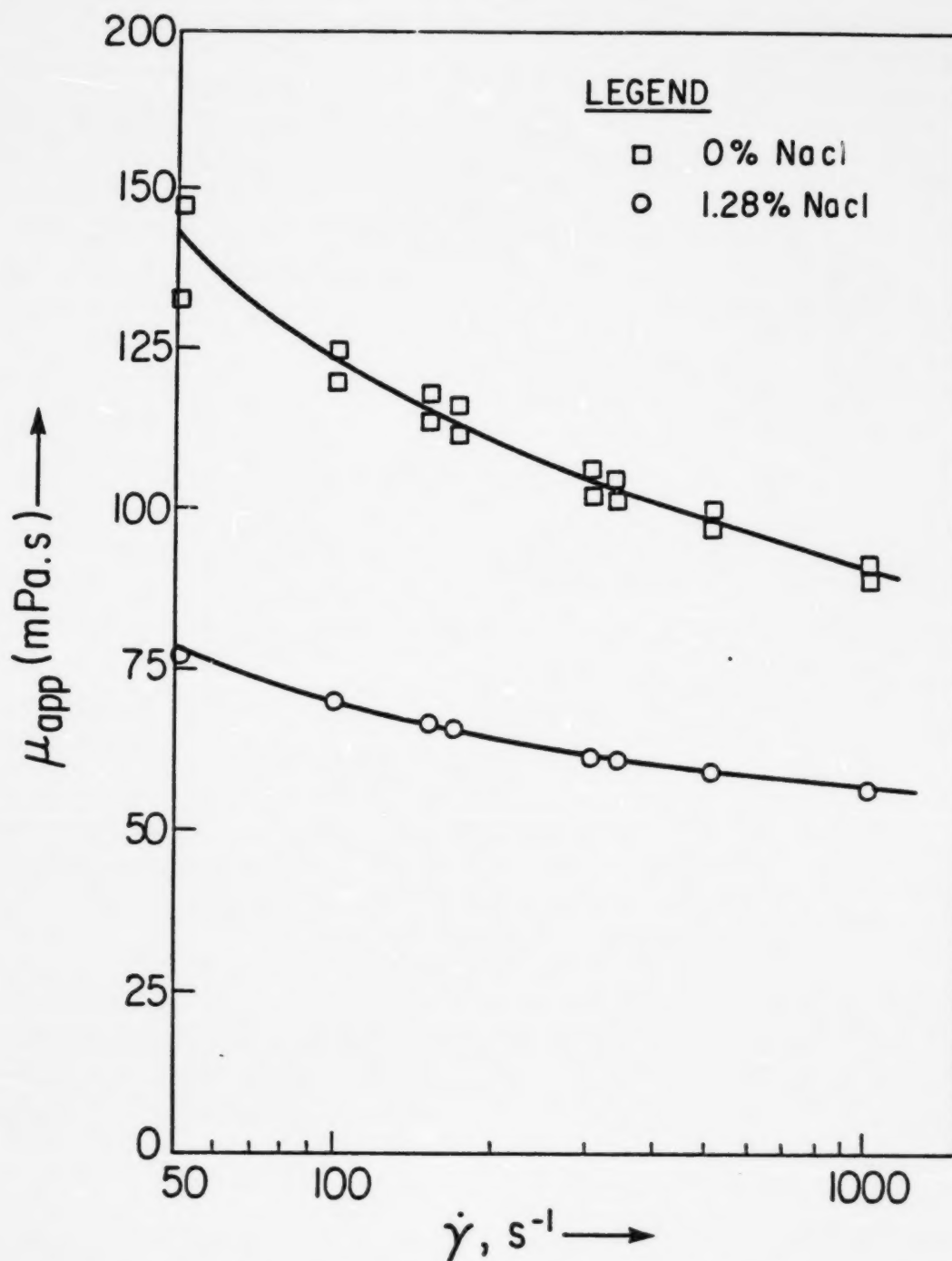


Figure 8. Flow curves of emulsions with different salt concentrations.

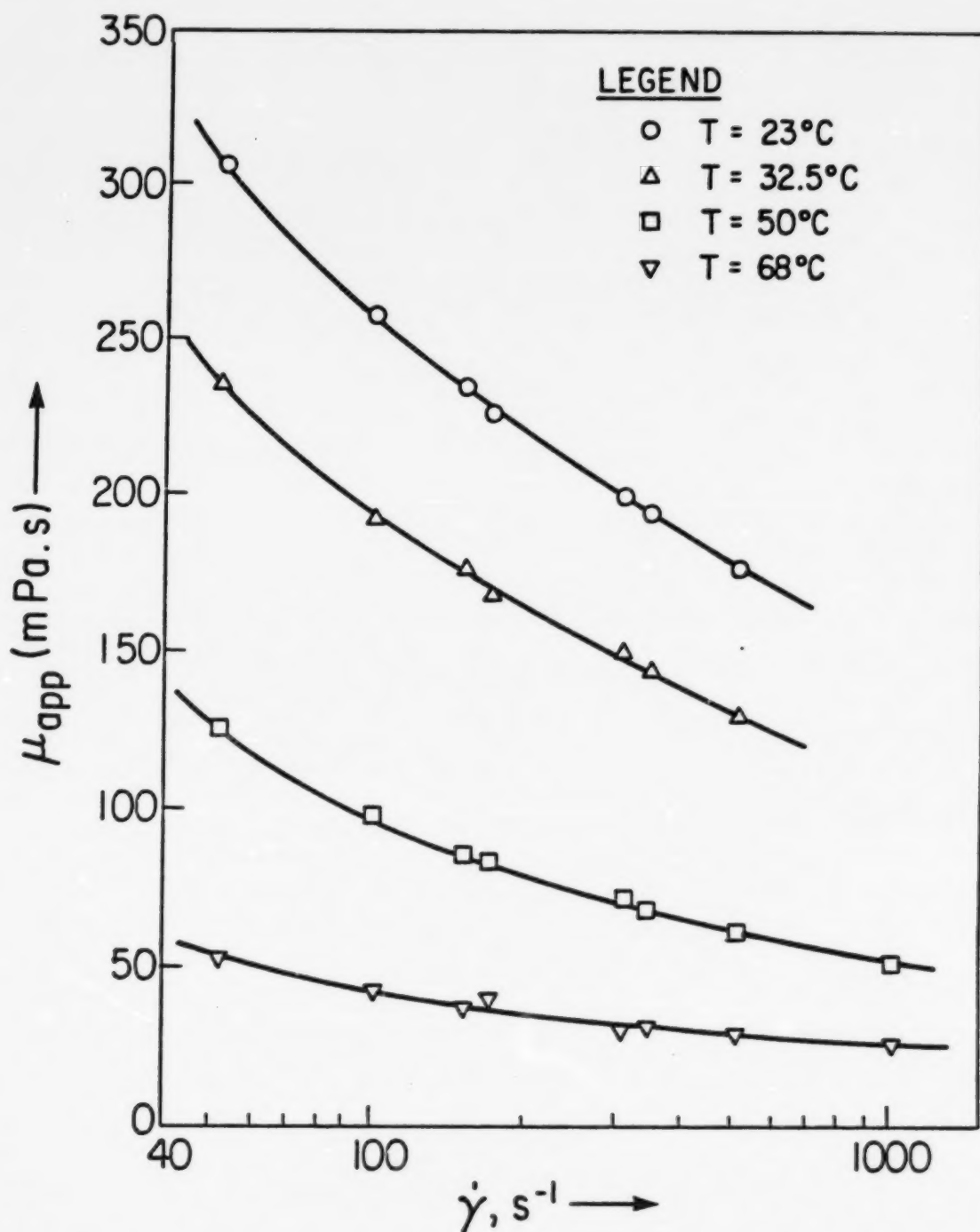


Figure 9. Flow curves of emulsions as affected by temperature.

conductivity meter. A set of experiments were run at various levels of concentration of Bayol/Water mixtures.

Using a mean density based on the oil/water concentration as measured by the conductivity meter, the orifice and venturi behaved in a very normal manner providing extremely good calibration curves for total mass flow. This performance extended to the situation where the emulsion inverted.

The magnetic flow meter also calibrated very well and of course had the added advantage of being independent of oil/water concentration. A drawback occurred when the phases inverted. Because the oil phase was non-conducting it was found that the magnetic flow meter gave no reading after inversion. It is recommended that the magnetic flow meter be further tested with bituminous oils because they do in fact have a conductivity and consequently, this may work in the magnetic flow meter. Certainly, the calibration curve is good when the continuous phase is conducting.

The conductance probe also calibrated for oil concentration when the continuous phase was conducting. When inversions occurred there was a sharp dip in the calibration curve making the use of the meter doubtful when the continuous phase is non-conducting. For bituminous oils this may not be the case and thus should be carefully studied.

A totally reliable and comprehensive metering system for all kinds of oil/water emulsions remains an elusive target for research engineers, although, the combination of the conductivity holdup meter with either the venturi, orifice or magnetic flow meter cannot yet be ruled out for bituminous oils.

APPENDIX 2

DATA ACQUISITION SOFTWARE

- b) Preliminary studies of a mineral oil/water emulsion using the Weissenbeijrg Rheogoniometer produced encouraging results. It was initially thought that the data would correlate well by using the so-called "maximum attainable concentration" as a parameter.
- c) Subsequent work using two other kinds of viscometers, namely the Fann and Brookfield types showed disparity in the data obtained from the three instruments. It was concluded that slip effects were affecting the correct results obtained from the Rheogoniometer and in subsequent work, the use of this instrument was abandoned.
- d) A broad spectrum of Rheological data was correlated using a new parameter $\phi_{\mu r=100}$ which is the concentration at which an emulsion viscosity becomes one hundred times that of the continuous phase. It is recommended that this correlation be tested on a wide variety of oil/water emulsions of both synthetic and produced fluids and that this method be used to predict the performance of emulsion pipelines.
- e) The Rheology of a real heavy oil (supplied by Petro-Canada from its facility at Cold Lake Alberta) was investigated using the Fann and Brookfield instruments. Based on the experimental data generated, the following conclusions were made:
 - The heavy oil alone behaves like a Newtonian fluid but changes viscosity with aging.
 - The O/W emulsion has a much higher viscosity than either of the single phases.

```

150 GOSUB 2000 'initialize channel numbers
160 L=1: R=0
160 INPUT "data from which pipe?";PIF.NUM
180 R=R+1
190 INPUT "pressure transducer channel?";PRE.CH#
190 Y=0: C=0: FLD=0
195 PRINT "PRESS ANY KEY TO CONT. "
195 ASK=INKEY$: IF ASK="" THEN 160%
100 CH.NO=PRE.CH# : GOSUB 4000
100 SUM PRE=SUM.PRE + READING(PRE.CH#)
150 Y=Y+1 : IF Y=1000 GOTO 1700
160 AVG.PRE=SUM.PRE/1000 'PRESSURE IN Pa.0
161 INPUT "flow rate units--if units=ml/sec then rate unit.flo=1---if units=kg/
to then unit.flo=2:UNIT.FLO
162 INPUT "flow rate in units specified above";RATE.FLO
163 IF UNIT.FLO=1 THEN LOW.O=RATE.FLO/10160
164 IF UNIT.FLO=2 THEN LOW.O=RATE.FLO/DEN.AVG
170 PRINT #1,USING"###.##### ";R;PIF.NUM;LOW.O;AVG.PRE
175 LPRINT USING"###.##### ";R;PIF.NUM;LOW.O;AVG.PRE
180 R=R+1: R=R+1: PRINT "no of flow rate runs taken=";R
190 INPUT "no of flow rate runs desired ";I
100 IF R<I GOTO 1590
10 L=L+1: IF L<5 GOTO 1560 'data from pipes 1,2,3,4 and 5
20 CLOSE #1: RETURN
30 ***** LOW FLOW SUB ENDS *****
100 ***** INITIALIZE CHANNEL NUMBERS SUB BEGINS *****
01
10
120 RESTORE 2080
30
40
50
60
30 DATA 0, CH 0, Test Data for Channel 0, V
31 DATA 1, CH 1, Test Data for Channel 1, V
32 DATA 2, CH 2, Test Data for Channel 2, V
33 DATA 3, CH 3, Test Data for Channel 3, V
34 DATA 4, CH 4, Test Data for Channel 4, V
35 DATA 5, CH 5, Test Data for Channel 5, V
36 DATA 6, CH 6, Test Data for Channel 6, V
37 DATA 7, CH 7, Test Data for Channel 7, V
38 DATA 8, CH 8, Test Data for Channel 8, V
39 DATA 9, CH 9, Test Data for Channel 9, V
40 DATA 10, CH 10, Test Data for Channel 10, V
41 DATA 11, CH 11, Test Data for Channel 11, V
42 DATA 12, CH 12, Test Data for Channel 12, V
43 DATA 13, CH 13, Test Data for Channel 13, V
44 DATA 14, CH 14, Test Data for Channel 14, V
45 DATA 15, CH 15, Test Data for Channel 15, V
46 DATA 16, CH 16, Output Channel 1, V
47 DATA 17, CH 17, Output Channel 2, V
48 DATA -1, " -1 Indicates end of data"
0
20 READ CH.NO
30 WHILE CH.NO >= 0
40 FOR I = 1 TO 3
50 READ CHAN#(CH.NO, I)
60 NEXT I
70 READ CH.NO
80 READ CH.NO
90 READ
100 RESTORE 2080
110
120
130
140
150
160
170
180
190
200
210
220
230
240
250
260
270
280
290
300
310
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Chapter 1

INTRODUCTION

Emulsions are mixtures of two immiscible liquids, one of them being dispersed in the other in the form of globules. There may exist either an oil-in-water (oil is the dispersed phase) or water-in-oil (water is the dispersed phase) emulsion. These emulsion types are conveniently abbreviated O/W and W/O, respectively. O/W and W/O emulsions are also sometimes sub-classified as being either "stable" or "unstable". Usually stable emulsions are formed with the aid of "emulsifiers" (surface active agents) or, as in the case of natural emulsions, with the help of salts and contaminants which also have significant surface active effects. Stable emulsions do not breakdown rapidly so that the phases separate. Unstable emulsions are usually formed from mixtures of pure substances (e.g. mineral oil and distilled water). The phases rapidly separate under the influence of gravity.

1.1 Naturally Produced Emulsions

A considerable amount of the world's crude oil is produced in the form of stable emulsions (1-7). In primary oil production, when a hole is drilled through the overlying impervious strata and the pressure on the reservoir is relieved in the immediate vicinity of the well bore, the higher pressure in the other portions of the reservoir causes the fluids to migrate to the bore hole. In so doing, they are forced through fine passages or pores in the rock, sometimes at considerable velocities, and ample opportunity is provided for the oil

```

10 ***** MAIN PROGRAM *****
20 GOTO 30
30 REMARKS : WITH 80 : COLOR 1,4,4
40 DIM C(20,3) : DIM L(20,4) : DIM R(20)
50 PRINT "TITLE OF EXP. MUST INCLUDE: DATE;TYPE OF EMULSION;DISP.PHASE;CONT.PH"
60 PRINT "STANDARD OF EMULSION PREPARATION"
70 PRINT "ENTERED TITLE OF EXP.:";TITLE$
80 INPUT "TANK FLUID TEMP IN DEG.C ";TEM.TAN
90 INPUT "ROOM TEMP IN DEG.C ";TEM.ROO
10 INPUT "CONT. PHASE WITH EMULSIFIER DENSITY IN Kg/m3 AT TANK TEMP";CONC.DEN
11 INPUT "CONT. PHASE WITH EMULSIFIER CONDUCTIVITY IN MILLI.MHO/M FROM KNOWN OF ";CONC.COND
12 INPUT "CONT. PHASE WITH EMULSIFIER CONDUCTANCE IN MILLIMHO FROM STAR CELL";CONC.STAR
13 INPUT "DISPERSE PHASE DENSITY AT TANK FLUID TEMP IN Kg/m3 ";DEN.DIS
14 INPUT "CONT. PHASE WITH Emulsifier viscosity at tank temp in PaS";VIS.CON
15 INPUT "DISPERSE PHASE VISCOSITY at tank temp in PaS";VIS.DIS
16 INPUT "Emulsion conductivity from known const. Cell at tank temp in milli.mh m";CONC.EMU
17 INPUT "EMULSION CONDUCTANCE FROM STAR CELL IN MILLIMHO";CONC.STAR
18 INPUT "volume % dispersed phase conc.";CONC.DIS
19 DEN.AVG=DEN.DIS*CONC.DIS + (1-CONC.DIS)*CONC.DEN
20 INPUT "HAVE YOU TAKEN RHEOLOGICAL DATA";YON$
21 LPRINT "TITLE OF EXPERIMENT IS ";TITLE$
22 LPRINT "TANK FLUID TEMP = ";TEM.TAN : LPRINT "ROOM TEMP. = ";TEM.ROO : LPRINT "DIS. PHASE DENSITY AT TANK TEMP. IN Kg/m3 = ";DEN.DIS : LPRINT "CONT.PHASE D . AT TANK TEMP. IN Kg/m3 = ";CONC.DEN
23 LPRINT "CONT.PHASE WITH EMULSIFIER VISCOSITY AT TANK FLUID TEMP. = ";VIS.CON
24 LPRINT "DISPERSE VISCOSITY AT TANK TEMP = ";VIS.DIS : LPRINT "CONT. WITH EMULSI ER CONDUCTIVITY AT TANK TEMP. IN MILLIMHO/M = ";CONC.COND
25 LPRINT "CONT. PHASE WITH EMULSIFIER CONDUCTANCE IN MILLIMHO FROM STAR CELL=" INT.STAR
26 LPRINT "VOL % CONC. OF DISPERSE PHASE= ";CONC.DIS : LPRINT "AVG. DENSITY OF JL. = ";DEN.AVG : LPRINT "EMUL. COND. THRU KNOWN CONSTANT CELL IN MILLIMHO/M = " CON.EMU : LPRINT "EMULSION CONDUCTANCE FROM STAR CELL IN MILLIMHO=";CONC.STAR
27 PRINT " PRESS ANY KEY TO CONT. "
28 A1$=INKEY$ : IF A1$="" THEN 236
29 PRINT "Z CAN HAVE 3 VALUES !!! FOR METER CALI. SUB. TAKE Z=1 , FOR LOW FLOW
30 . TAKE Z=2 , FOR HIGH FLOW SUB. TAKE Z=3 "
31 INPUT "VALUE OF Z";Z
32 IF Z=1 GOTO 281 'METER CALIBRATION SUBROUTINE
33 IF Z=2 GOTO 283 'LOW FLOW SUBROUTINE
34 IF Z=3 GOTO 285 'HIGH FLOW SUBROUTINE
35 GOSUB 9000 'METER CALIBRATION SUB
36 GOTO 310
37 GOSUB 1500 'LOW FLOW SUB
38 GOTO 310
39 GOSUB 5000 'HIGH FLOW SUB
40 GOTO 310
41 PRINT " PRESS ANY KEY TO CONT. "
42 A2$=INKEY$ : IF A2$="" THEN 320
43 GOTO 240
44 ***** MAIN PROGRAM ENDS *****
45 ***** LOW SUB REGIMS *****
46 ***** LOW SUB REGIMS *****
47 ***** LOW SUB REGIMS *****
48 ***** LOW SUB REGIMS *****
49 ***** LOW SUB REGIMS *****
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57 ***** LOW SUB REGIMS *****
58 ***** LOW SUB REGIMS *****
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96 ***** LOW SUB REGIMS *****
97 ***** LOW SUB REGIMS *****
98 ***** LOW SUB REGIMS *****
99 ***** LOW SUB REGIMS *****
100 ***** LOW SUB REGIMS *****

```

Many situations exist where these emulsions have to be transported long distances by pipeline from production centres to dehydration centres etc. Accurate metering and prediction of flow behaviour of these emulsions is, therefore, extremely important for royalty calculations, estimation of the capacity of existing oil pipelines and design of new pipelines.

1.2 Deliberately Produced Emulsions

The high viscosity of heavy oils or produced W/O emulsions frequently limits the rate at which crude oil can be produced from a well. It causes low pump volumetric efficiency, slow rod drop rate and high flow line pressure drop. To overcome these problems (8-12), aqueous solutions of surface active chemicals (non-ionic surfactants or soaps) are sometimes added directly to the well bore to convert highly viscous oil or W/O emulsion to low viscosity O/W emulsion.

The transportation of highly viscous crude oils (especially high pour point waxy crudes) in the form of concentrated O/W emulsions has also received special attention in the recent years (13-30). The viscosity of crude oils is greatly reduced by forming O/W emulsions. For example, the viscosity of pure Boscan crude is 80,000 cp. at 20°C. However, the viscosity of an emulsion containing 75 percent Boscan crude and 25 percent water is only 50 cp. at 20°C. Emulsion transport eliminates the need to keep viscous crude hot during transport and avoids the problem of "freeze-up" in cases of flow stoppage. Another important application for emulsion pumping is in


```

166
167 DATA 0, 0.0, 10, 0.0, 10.0, -1.0, 11.0
168 DATA 1, 0.0, 10, 0.0, 10.0, -1.0, 11.0
169 DATA 2, 0.0, 10, 0.0, 10.0, -1.0, 11.0
170 DATA 3, 0.0, 10, 0.0, 10.0, -1.0, 11.0
171 DATA 4, 0.0, 10, -79.136584, 739.87258, -1.0, 11.0
172 DATA 5, 0.0, 10, -348.06426, 2665.8713, -1.0, 11.0
173 DATA 6, 0.0, 10, -756.46781, 13235.212, -1.0, 11.0
174 DATA 7, 0.0, 10, 0.0, 10.0, -1.0, 11.0
175 DATA 8, 0.0, 10, 0.0, 10.0, -1.0, 11.0
176 DATA 9, 0.0, 10, 0.0, 10.0, -1.0, 11.0
177 DATA 10, 0.0, 10, 0.0, 10.0, -1.0, 11.0
178 DATA 11, 0.0, 10, 0.0, 10.0, -1.0, 11.0
179 DATA 12, 0.0, 10, -2333.8491, 33873.56, -1.0, 11.0
180 DATA 13, 0.0, 10, -5485.072, 83428.613, -1.0, 11.0
181 DATA 14, 0.0, 10, -11843.756, 225104.18, -1.0, 11.0
182 DATA 15, 0.0, 10, 0.0, 10.0, -1.0, 11.0
190 DATA -1,
                                " -1 Indicates end of data"
200 READ CH.NO
202 WHILE CH.NO >= 0
203     READ X1, X2, Y1, Y2
209     SLOPE = (Y2 - Y1) / (X2 - X1)
210     CHAN(CH.NO, 1) = SLOPE
215     CHAN(CH.NO, 2) = Y1 - SLOPE * X1
218     READ CHAN(CH.NO, 3), CHAN(CH.NO, 4)
219     READ CH.NO
220 WEND
230
240 Range Setting
260     input range : 0 to +10 volts
274     input gain : FGL (Low inputs: 1,10,100,500)
299 RETURN
300 ***** INITIALIZE CHANNEL NUMBERS SUB ENDS *****
300 ***** A/D READING SUB BEGINS *****
320 *****
330 ***** TECHMAR LABMASTER A/D Sampler *****
340
350 Inputs : CH.NO the channel you wish to sample
360 Outputs : READING the channel read (in final units)
370 ERR.NO set when any errors are detected
380
390 GAINX=0
400 LM.BASE = &H710
410 OUT LM.BASE+4, 128+GAINX
420 OUT LM.BASE+5, CH.NO
430 OUT LM.BASE+6, 0
440 IF INP(LM.BASE+4) < 128 THEN 4130
450 LOW.BYTEX = INP(LM.BASE+5)
460 HIGH.BYTEX = INP(LM.BASE+6)
470 VAR = LOW.BYTEX + HIGH.BYTEX * 256
480 IF HIGH.BYTEX > 1 AND HIGH.BYTEX < 128 THEN 4170
490 IF HIGH.BYTEX < 255 AND HIGH.BYTEX > 127 THEN 4170
500
510 Convert per cent full scale to final units
520
530 VAR = VAR / 2
540
550 VAR = VAR/10^(GAINX)
560 IF GAINX = 3 THEN VAR = VAR*2
570 VOLTAGE = VAR / 204.8
580 READING(CH.NO) = VOLTAGE * CHAN(CH.NO, 1) + CHAN(CH.NO, 2)
590 RETURN
600 ***** A/D READING SUB ENDS *****
610 ***** HIGH FLOW SUB BEGINS *****
620 ***** HIGH FLOW SUB - HIGH FLOW SUB DATA AND MEASUREMENT DATA COLLECTION *****

```

The work described in this report represents the beginnings of an attempt to address the problems of O/W and W/O emulsion rheological classification as well as the problem of O/W and W/O emulsion metering. During the course of the work the immense complexity of the emulsion problem has become more and more apparent to the investigators. Many questions remain to be posed and answered. Some new ideas for generalizing the rheological results have been suggested and a possible system of instrumentation has been investigated.

At the outset of the work the following "tasks" were envisaged:

1. Modification and assembly of flow loop.
2. Development of a micro-computer data acquisition system for flow loop.
3. Development of data acquisition software.
4. Survey and acquisition of representative oils.
5. Running of experiments - rheological and flow loop.
6. Evaluation of experiments and flow meters.
7. Development of multiphase flow meters.
8. Final report.

This report contains results pertaining to all of the above tasks. At the beginning of each chapter in the report there is an indication as to which of the above tasks are being reported either in whole or in part.


```

025 K4="PIF.NUM":N4="PIF.FRE" : N4="AVG.MAG": O4="AVG.ORI" : O4="READ.NO"
030 WRITE #1,TITLE# : LPRINT "HIGH FLOW DATA"
035 PRINT#1,USING"\          \";O4;K4;M4;N4;O4
036 LPRINT USING"\          \";O4;K4;M4;N4;O4
040 GOSUB 2000 'initialize channel numbers
045 DIM A(3) : L=0 : K=0 : R=0
100 INPUT "MAG FLOW METER CHANNEL"; MAG.CHA
110 A(1)=MAG.CHA
135 INPUT "DATA FROM METERING SECTION AND WHICH PIPE ??";PIF.NUM
140 INPUT "PRESSURE CHANNEL FOR PIPE ";PIF.CHA
150 A(2)=PIF.CHA
160 INPUT "PRESSURE CHANNEL FOR ORIFICE"; ORI.CHA
170 A(3)=ORI.CHA
190 P=0 : SUMPIF.CHA=0
200 SUMORI.CHA=0 : SUMMAG.CHA=0
234 PRINT " PRESS ANY KEY TO CONT. "
235 A4$=INKEY$ : IF A4$="" THEN 5235
240 NO.CHA=3 : FOR V=1 TO NO.CHA : CH.NO=A(V)
250 GOSUB 4000 'READ GIVEN CHANNEL
260 NEXT V
300 SUMPIF.CHA=SUMPIF.CHA + READING(PIF.CHA)
310 SUMORI.CHA=SUMORI.CHA + READING(ORI.CHA)
320 SUMMAG.CHA=SUMMAG.CHA + READING(MAG.CHA)
360 P=P+1 : IF P<1000 GOTO 5240
390 AVGPRES.PIF=SUMPIF.CHA/1000 'Pa.S
400 AVG.ORI=SUMORI.CHA/1000 'Pa.S
420 AVG.MAG=SUMMAG.CHA/1000 'VOLTS
440 PRINT #1,USING "###.####^#### \";R;PIF.NUM;AVGPRES.PIF;AVG.MAG;AVG.ORI
46 LPRINT USING "###.####^#### \";R;PIF.NUM;AVGPRES.PIF;AVG.MAG;AVG.ORI
50 L=L+1 : R=R+1 : PRINT "NO OF FLOW RATE RUNS ALREADY TAKEN ";L
60 INPUT "NO OF FLOW RATE RUNS DESIRED ";T
70 IF L<T GOTO 5140
80 K=K+1 : IF K<3 GOTO 5490 ' data only from 3 pipes namely 3,4 and 5
81 CLOSE#1 : RETURN
90 L=0 : GOTO 5135
00 '***** HIGH FLOW SUB ENDS *****
00 '***** METER CALIBRATION SUB BEGINS *****
00 OPEN "B:CALIBRAT.Dat" FOR APPEND AS#1
20 INPUT "BRIEF title of expt."; TITLE#
25 A$="READ.NO" : B$="MAS.FLO" : C$="AVG.MAG" : D$="AVG.ORI" : E$="AVG.VEN"
30 WRITE #1,TITLE# : LPRINT "METER CALIBRATION DATA"
35 PRINT#1,USING"\          \";A$,B$,C$,D$,E$
36 LPRINT USING"\          \";A$,B$,C$,D$,E$
40 GOSUB 2000 'initialize channel numbers
45 DIM R(3) : L=0 : K=0
60 INPUT "MAG FLOW METER CHANNEL"; MAG.CHA
70 R(1)=MAG.CHA
80 INPUT "PRESSURE CHANNEL FOR ORIFICE"; ORI.CHA
90 R(2)=ORI.CHA
90 INPUT "PRESSURE CHANNEL FOR VENTURI ";VEN.CHA
45 R(3)=VEN.CHA
50 P=0
60 SUMORI.CHA=0 : SUMVEN.CHA=0 : SUMMAG.CHA=0 :
95 PRINT " PRESS ANY KEY TO CONT. "
06 A5$=INKEY$ : IF A5$="" THEN 9206
10 NO.CHA=3 : FOR T=1 TO NO.CHA : CH.NO=R(T)
10 GOSUB 4000 'READ GIVEN CHANNEL
20 NEXT T
30 SUMORI.CHA=SUMORI.CHA + READING(ORI.CHA)
40 SUMVEN.CHA=SUMVEN.CHA + READING(VEN.CHA)
50 SUMMAG.CHA=SUMMAG.CHA + READING(MAG.CHA)
60 P=P+1 : IF P<500 GOTO 9210
70 AVG.ORI=SUMORI.CHA/500 'Pa.S
80 AVG.VEN=SUMVEN.CHA/500 'Pa.S
90 AVG.MAG=SUMMAG.CHA/500 'VOLTS

```

Little published information is available on turbulent flow behaviour of stable O/W emulsions. Only one reference (30) is available in which studies on turbulent flow of stable O/W emulsions is reported. However, the results of this study have not been correlated successfully. Cengel, et. al (56) studied laminar and turbulent flow of dilute (oil concentration less than 50 percent) unstable oil-in-water emulsions. Assuming that the dispersion behaved as a single phase fluid, effective viscosities of the mixtures were calculated from measured friction factors. In laminar flow, viscosities were found to be a function of capillary diameter, possibly due to the formation of a thin layer of continuous phase adjacent to the capillary wall. Up to 20% oil, the viscosity of the dispersions was the same in laminar, vertical turbulent and horizontal turbulent flow. In vertical turbulent flow, all dispersions behaved as Newtonian fluids. The 35 and 50 percent dispersions in horizontal flow exhibited non-Newtonian characteristics and had effective viscosities considerably less than the same dispersion in vertical flow. The apparent non-Newtonian behaviour could be due to phase separation in the horizontal flow.

Zakin et. al, (30) studied the turbulent flow behaviour of O/W emulsions stabilized with Triton X-114 (non-ionic emulsifier). Turbulent flow pressure drop measurements were made for various concentrated O/W emulsions which exhibited non-Newtonian behaviour. Measured friction factors consistently fell below those predicted by the Dodge and Metzner friction factor correlation. No further attempt was made to correlate the friction factor data. It was suggested that the departure from the Dodge and Metzner correlation could be due to:

```
355 INPUT FLOW RATE (IN KG/SEC); MASS.FLO
360 PRINT#1, USING "###.##### "; L, MASS.FLO, AVG. MAG, AVG. ORI, AVG. VEN
365 LPRINT USING "###.##### "; L, MASS.FLO, AVG. MAG, AVG. ORI, AVG. VEN
370 L=L+1 : PRINT "NO OF FLOW RATE RUNS ALREADY TAKEN "; L
380 INPUT "NO OF FLOW RATE RUNS DESIRED "; T
390 IF L < T GOTO 3120
395 CLOSE#1
400 RETURN
410 ***** METER CALIBRATION SUB ENDS *****
```

Chapter 3

FUNDAMENTALS OF NON-NEWTONIAN FLOW

The theory reviewed in this Chapter relates to the results obtained in Tasks 4 and 5 of the contract.

3.1 Steady-State Laminar Flow of Time-Independent Non-Newtonian Fluids (57-63)

A perfectly general relationship for the steady, laminar flow of any time-independent purely viscous fluid may be developed from a generalized constitutive equation. The resulting equations are usually referred to as Rabinowitsch-Mooney relations.

We start from a constitutive equation which, however complex, may be expressed as

$$f(\tau) = \gamma \quad (3.10)$$

where

τ = shear stress

γ = absolute value of shear rate = $-du/dr$ in a circular pipe.

For steady flow in a pipe

$$Q = 2\pi \int_0^R u r dr \quad (3.11)$$

```

000 *****
010 P Plotting Routine for Graphics Monitor
020
030
040 plots curves of data in the X-Y co-ordinates
050
060 Required
070 x.pts() . . . . . x values
080 y.pts() . . . . . y values
090 N . . . . . number of points to be plotted
100
110 Optional
120 TITLE$ . . . . . title of graph (max = 35 chars)
130 X$ . . . . . x-axis label (max = 35 chars)
140 Y$ . . . . . y-axis label (max = 20 chars)
150 CONNECT . . . . . = -1 if points are to be connected
160 RE.PLOT . . . . . = -1 if plot is to be added to graph
170 GRID . . . . . = -1 if a grid is to be drawn
180 XMIN . . . . . minimum x-axis value
190 XMAX . . . . . maximum x-axis value
200 YMIN . . . . . minimum y-axis value
210 YMAX . . . . . maximum y-axis value
220
230 Returned
240 ERR.NO . . . . . error encountered
250 1 = zero length axis
260 2 = error detected in subroutine
270 save program and consult J.Allen
280 Variables
290 I . . . . . general counter
300 ORIGIN$ . . . . . origin of graph
310 PLOT.COLOR . . . . . color of current plot
320 TICK$ . . . . . location of next tick mark
330 X . . . . . next x value to plot in screen units
340 X.EXP% . . . . . log(x) base 10
350 X.SCALE . . . . . X (screen units) = X.pts() * X.SCALE
360 Y . . . . . next x value to plot in screen units
370 Y.EXP% . . . . . log(x) base 10
380 Y.SCALE . . . . . Y (screen units) = Y.pts() * Y.SCALE
390
95 NOTE: If the scale on the axes is not specified (via XMAX XMIN YMAX YMIN)
96 then the subroutine calculates them by scanning the array of data.
97 If the routine is called initially with a single data point then
98 it is impossible for the routine to determine a scale. The scale
99 must be specified by the caller if a single point is used.
00 DEF FNLOG10(X) = LOG(X)/LOG(10) ' log for base 10
10 IF RE.PLOT THEN GOTO 7100 ' skip screen setup
20 CLS
30 SCREEN 1,0,0,0
40 ORIGIN$ = "C2 BM49,176"
50 KEY OFF
60 COLOR 0,0
70
80 Find Limits of axes
90
00 IF XMAX <> XMIN THEN GOTO 6570 ' user chosen X axis
10 XMAX = XMIN = X.PTS(1)
20 FOR I = 1 TO N
30 IF XMAX < X.PTS(I) THEN XMAX = X.PTS(I)
40 IF XMIN > X.PTS(I) THEN XMIN = X.PTS(I)
50 NEXT I

```

$$r^2 = \frac{R^2}{\tau_w^2} \tau^2 \quad \text{and} \quad dr = \frac{R}{\tau_w} d\tau$$

Combining Eqs. (3.10) and (3.13) and substituting the foregoing for r^2 and dr , we obtain

$$Q = \pi \int_0^{\tau_w} \frac{R^3 \tau^2}{\tau_w^3} f(\tau) d\tau \quad (3.17)$$

If the value of τ_w is inserted in equation (3.17) and R is replaced by $D/2$, the following equation results.

$$\frac{8Q}{\pi D^3} = \frac{2V}{D} = \frac{1}{\left(\frac{D\Delta P}{4L}\right)^3} \int_0^{\frac{D\Delta P}{4L}} \tau^2 f(\tau) d\tau \quad (3.18)$$

Where L denotes the length of the pipe. This is a perfectly general relationship of the discharge Q , the pressure gradient $\Delta P/L$, the diameter D , and the constitutive equation $f(\tau) = -du/dr$. Equation (3.18) may be rearranged and differentiated with respect to $D\Delta P/4L$ to yield

$$f(\tau_w) = - \left(\frac{du}{dr} \right)_w = 3 \left(\frac{8Q}{\pi D^3} \right) + \frac{D\Delta P}{4L} \frac{d \left(\frac{8Q}{\pi D^3} \right)}{d \left(\frac{D\Delta P}{4L} \right)} \quad (3.19)$$


```

360 IF YMAX <> YMIN THEN GOTO 6630 'user chosen Y axis
370 YMAX = YMIN = Y.PTS(1)
380 FOR I = 1 TO N
390 IF YMAX < Y.PTS(I) THEN YMAX = Y.PTS(I)
400 IF YMIN > Y.PTS(I) THEN YMIN = Y.PTS(I)
410 NEXT I
420
430 PLOT axes
440
450 DRAW ORIGIN# ' move to origin
460 DRAW "U160 L7 d165 R267 U5 L260" ' outline axes
470 PAINT STEP(-1,-1),2,2 ' fill in axes
480
490 Plot Scale on Axes
500
510 FOR I = 0 TO 10 'along x axis
520 X = I * 24 + 50
530 IF GRID THEN TICK$ = "BM" + STR$(X) + ",15 M" + STR$(X) + ",190"
540 ELSE TICK$ = "BM" + STR$(X) + ",180 M" + STR$(X) + ",190"
550 DRAW TICK$
560 NEXT I
570
580 FOR I = 0 TO 10 'along y axis
590 Y = 175 - I * 16
600 IF GRID THEN TICK$ = "BM37," + STR$(Y) + " M290," + STR$(Y)
610 ELSE TICK$ = "BM37," + STR$(Y) + " M42," + STR$(Y)
620 DRAW TICK$
630 NEXT I
640
650 Put Scale on axes
660
670 IF XMAX-XMIN = 0 OR YMAX-YMIN = 0 THEN ERR.NO = 2: RETURN
680 X.EXP% = FNLOG10(XMAX-XMIN)
690 Y.EXP% = FNLOG10(YMAX-YMIN)
700 FOR I = 0 TO 5
710 LOCATE 24, 6*I + 5
720 PRINT USING"###.#"; (XMIN + I*((XMAX-XMIN)/5))/ 10^X.EXP%;
730 LOCATE 22 - I*4,2
740 PRINT USING"###.#"; (YMIN + I*((YMAX-YMIN)/5))/ 10^Y.EXP%;
750 NEXT I
760 LOCATE 25,34: PRINT "E";:PRINT USING "###";X.EXP%;
770 LOCATE 23,1 : PRINT "E";:PRINT USING "###";Y.EXP%;
780
790 Plot Titles
800
810 TITLE$ = MID$(TITLE$,1,35) ' truncate titles
820 X$ = MID$(X$,1,35)
830 Y$ = MID$(Y$,1,20)
840 LOCATE 1,(20-INT(LEN(TITLE$)/2)): PRINT TITLE$;
850 LOCATE 25,(20-INT(LEN(X$)/2)): PRINT X$;
860 CHARS = (12-INT(LEN(Y$)/2))
870 FOR I = 1 TO LEN(Y$)
880 LOCATE I + CHARS, 1
890 PRINT MID$(Y$,I,1);
900 NEXT I
910
920 Plot Data Points
930
940 IF XMAX-XMIN = 0 OR YMAX-YMIN = 0 THEN ERR.NO = 1: RETURN 'bad scales
950 X.SCALE = (XMAX - XMIN) / (310 - 70)
960 Y.SCALE = (YMAX - YMIN) / (175 - 15)
970 PLOT.COLOR = PLOT.COLOR + 1
980 IF PLOT.COLOR > 3 THEN PLOT.COLOR = 1
990 FOR I = 1 TO N
1000 X = X.PTS(I): Y = Y.PTS(I)

```


be drawn by repeating this procedure at different values of the wall shear stress. If the slope of the logarithmic plot is constant over a range of shear stresses, Eqn. (3.112) represents the equation of this straight line. If the plot is not a straight line, n' and K' must be evaluated at the appropriate shear stress.

For a power law pseudoplastic, it can be easily shown that

$$n' = n \quad k' = k \left(\frac{1 + 3n}{n} \right) \quad (3.113)$$

Where n and K are power law constants ($\tau = K \dot{\gamma}^n$).

Friction Factor Versus Reynolds Number for Non-Newtonian Fluids

The friction factor, f , is defined in the usual manner by

$$f = \frac{\tau_w}{\frac{1}{2}(\rho V^2)} = \frac{D \Delta p}{4L} / \frac{\rho \bar{V}^2}{2} \quad (3.114)$$

In order to be able to use the conventional chart for Newtonian fluids and non-Newtonian fluids alike in the laminar region, it is convenient to let $f = 16/N'_{Re}$ as for Newtonians in laminar flow. Since

$$\tau_w = \frac{D \Delta p}{4L} = K' \left(\frac{8\bar{V}}{D} \right)^{n'} \quad (3.112)$$

```

200 IF X < XMIN THEN X = XMIN
210 IF X > XMAX THEN X = XMAX
220 IF Y < YMIN THEN Y = YMIN
230 IF Y > YMAX THEN Y = YMAX
240 X = 50 + INT( (X - XMIN) / X.SCALE )
250 Y = 175 - INT( (Y - YMIN) / Y.SCALE )
260 IF NOT CONNECT THEN PSET(X,Y),PLOT.COLOR:GOTO 7300
270 IF CONNECT AND I > 1 THEN LINE (X.OLD,Y.OLD)-(X,Y),PLOT.COLOR
280 X.OLD = X
290 Y.OLD = Y
300 NEXT I
310 RETURN
=====

```

If there is no anomalous behaviour or effective slip near the tube wall, $Q/\pi R^3$ versus $\Delta P/4L$ data will result in a single curve for different tube diameters. However, if there is effective slip near the tube wall, the curves will be distinct for each value of R .

3.3 Steady-State Turbulent Flow of Time-Independent Non-Newtonian Fluids (80-93)

Dodge and Metzner (80) carried out a semi-theoretical analysis of turbulent flow in smooth tubes of time-independent viscous non-Newtonian fluids described by the power law. Applying the techniques of dimensional analysis for the power law fluid, as Millikan (81) has done for Newtonian fluids, they derived the expression

$$\frac{1}{\sqrt{f}} = A_{1n} \log [N'_{Re} f^{\frac{(1-n')}{2}}] + C'_n \quad (3.30)$$

where

$$C'_n = A_{1n} \log \left[\frac{1}{8} \left(\frac{3 + 6n'}{n'} \right) n' \right] + C_n$$

The terms A_{1n} and C'_n are functions of n' to be determined experimentally. If n' is taken as unity, Eqn. (3.30) reduces to

$$\frac{1}{\sqrt{f}} = A \log \left(\frac{\rho D V}{\mu} \sqrt{f} \right) + C \quad (3.31)$$

APPENDIX 2

DATA ACQUISITION SOFTWARE

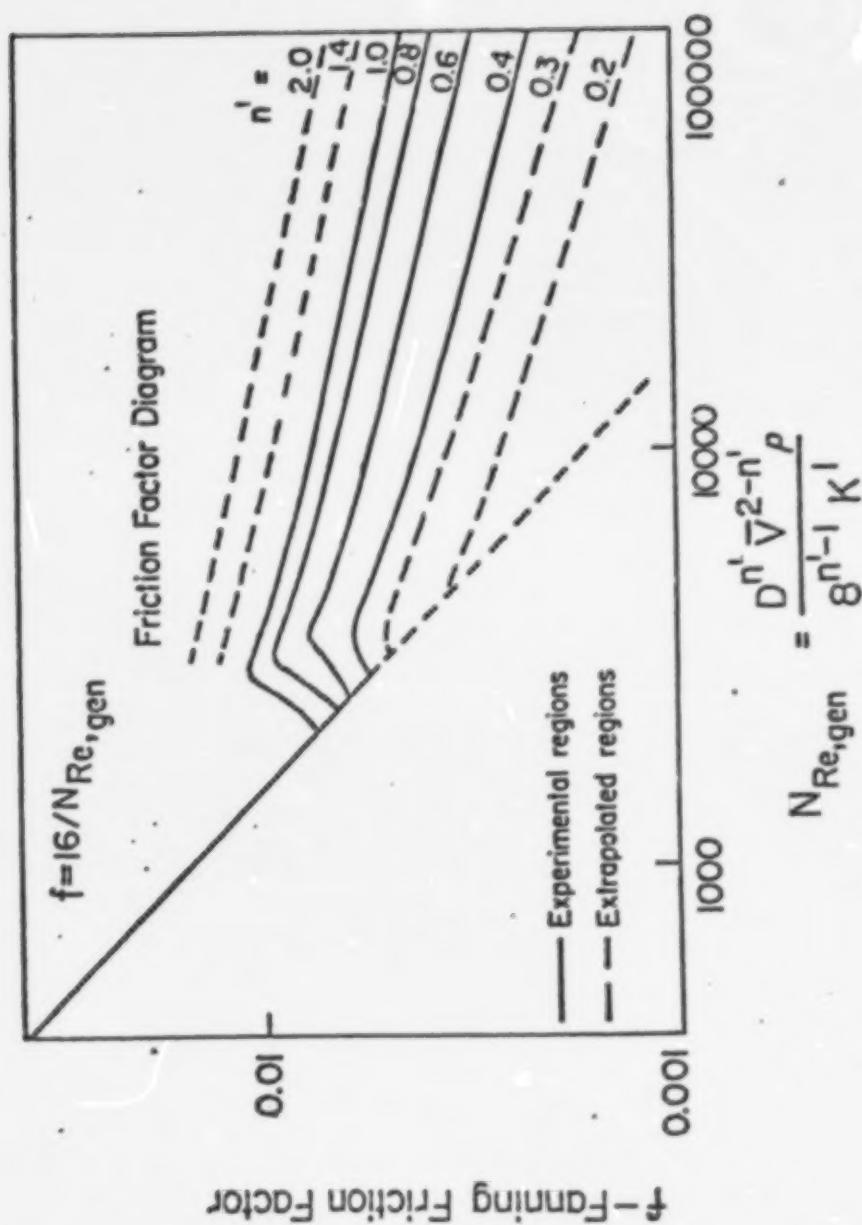


Figure 3.1: Friction Factor Diagram for Non-Newtonian Flow as Suggested by Dodge and Metzner (80).

```

1. CLR
2. DIMENSION: WITHIN 500: LOCUS 7,4,4
3. DIM CONCENT: 20,3: DIM CONDENS: 4: DIM READINGS(20)
4. PRINT "TITLE OF EXP. MUST INCLUDE: DATE; TYPE OF EMULSION; DISP. PHASE; CONT. PH"
5. "EMULSIFICATION METHOD OF EMULSION PREPARATION"
6. INPUT "ENTER DETAILED TITLE OF EXPT. "; TITLE#
7. INPUT "TANK FLUID TEMP IN DEG.C "; TEM.TAN
8. INPUT "ROOM TEMP IN DEG.C "; TEM.ROO
9. INPUT "CONT. PHASE WITH EMULSIFIER DENSITY IN Kg/m3 AT TANK TEMP; CONT.DEN
10. INPUT "CONT. PHASE WITH EMULSIFIER CONDUCTIVITY IN MILLI.MHO/M FROM KNOWN CO"
11. "CONT.COND
12. INPUT "CONT. PHASE WITH EMULSIFIER CONDUCTANCE IN MILLIMHO FROM STAR CELL"; C
13. STAR
14. INPUT "DISPERSE PHASE DENSITY AT TANK FLUID TEMP IN Kg/m3 "; DEN.DIS
15. INPUT "CONT. PHASE WITH Emulsifier viscosity at tank temp in PaS"; VIS.CON
16. INPUT "DISPERSE PHASE VISCOSITY at tank temp in PaS"; VIS.EDIS
17. INPUT "Emulsion conductivity from known const. Cell at tank temp in milli.mh"
18. "m"; COND.EMU
19. INPUT "EMULSION CONDUCTANCE FROM STAR CELL IN MILLIMHO"; COND.STAR
20. INPUT "volume % dispersed phase conc."; CONC.DIS
21. DEN.AVG=DEN.DIS*CONC.DIS + (1-CONC.DIS)*CONT.DEN
22. INPUT "HAVE YOU TAKEN RHEOLOGICAL DATA"; YON#
23. LPRINT "TITLE OF EXPERIMENT IS "; TITLE#
24. LPRINT "TANK FLUID TEMP = "; TEM.TAN : LPRINT "ROOM TEMP. = "; TEM.ROO : LPRIN
25. "DIS. PHASE DENSITY AT TANK TEMP. IN Kg/m3 = "; DEN.DIS : LPRINT "CONT.PHASE D
26. "AT TANK TEMP. IN Kg/m3 = "; CONT.DEN
27. LPRINT "CONT.PHASE WITH EMULSIFIER VISCOSITY AT TANK FLUID TEMP. = "; VIS.CON
28. LPRINT "DISPERSE VISCOSITY AT TANK TEMP = "; VIS.DIS : LPRINT "CONT. WITH EMULSI
29. "R CONDUCTIVITY AT TANK TEMP. IN MILLIMHO/M = "; CONT.COND
30. LPRINT "CONT. PHASE WITH EMULSIFIER CONDUCTANCE IN MILLIMHO FROM STAR CELL="
31. "NT.STAR
32. LPRINT "VOL % CONC. OF DISPERSE PHASE= "; CONC.DIS : LPRINT "AVG. DENSITY OF
33. "L. = "; DEN.AVG : LPRINT "EMUL. COND. THRU KNOWN CONSTANT CELL IN MILLIMHO/M= "
34. "ID.EMU : LPRINT "EMULSION CONDUCTANCE FROM STAR CELL IN MILLIMHO="; COND.STAR
35. LPRINT "PRESS ANY KEY TO CONT. "
36. A1#=INKEY$: IF A1#="" THEN 236
37. PRINT "Z CAN HAVE 3 VALUES !!! FOR METER CALI. SUB. TAKE Z=1 , FOR LOW FLOW
38. "TAKE Z=2 , FOR HIGH FLOW SUB. TAKE Z=3 "
39. INPUT "VALUE OF Z"; Z
40. IF Z=1 GOTO 281 'METER CALIBRATION SUBROUTINE
41. IF Z=2 GOTO 283 'LOW FLOW SUBROUTINE
42. IF Z=3 GOTO 285 'HIGH FLOW SUBROUTINE
43. GOSUB 9000 'METER CALIBRATION SUB
44. GOTO 310
45. GOSUB 1500 'LOW FLOW SUB
46. GOTO 310
47. GOSUB 5000 'HIGH FLOW SUB
48. GOTO 310
49. PRINT "PRESS ANY KEY TO CONT. "
50. A2#=INKEY$: IF A2#="" THEN 320
51. GOTO 240
52. ***** MAIN PROGRAM ENDS *****
53. ***** LOW SUB BEGINS *****
54. "Flow stop and start - Allow water and low flow to settle
55. "USE THIS AND LOW FLOW FOR APPROX 1 MIN
56. "Enter title of exp. "; TITLE#
57. "Flow stop and start - Allow water and low flow to settle
58. "USE THIS AND LOW FLOW FOR APPROX 1 MIN
59. "Flow stop and start - Allow water and low flow to settle
60. "USE THIS AND LOW FLOW FOR APPROX 1 MIN
61. "Flow stop and start - Allow water and low flow to settle
62. "USE THIS AND LOW FLOW FOR APPROX 1 MIN
63. "Flow stop and start - Allow water and low flow to settle
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67. "Flow stop and start - Allow water and low flow to settle
68. "USE THIS AND LOW FLOW FOR APPROX 1 MIN
69. "Flow stop and start - Allow water and low flow to settle
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71. "Flow stop and start - Allow water and low flow to settle
72. "USE THIS AND LOW FLOW FOR APPROX 1 MIN
73. "Flow stop and start - Allow water and low flow to settle
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75. "Flow stop and start - Allow water and low flow to settle
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81. "Flow stop and start - Allow water and low flow to settle
82. "USE THIS AND LOW FLOW FOR APPROX 1 MIN
83. "Flow stop and start - Allow water and low flow to settle
84. "USE THIS AND LOW FLOW FOR APPROX 1 MIN
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88. "USE THIS AND LOW FLOW FOR APPROX 1 MIN
89. "Flow stop and start - Allow water and low flow to settle
90. "USE THIS AND LOW FLOW FOR APPROX 1 MIN
91. "Flow stop and start - Allow water and low flow to settle
92. "USE THIS AND LOW FLOW FOR APPROX 1 MIN
93. "Flow stop and start - Allow water and low flow to settle
94. "USE THIS AND LOW FLOW FOR APPROX 1 MIN
95. "Flow stop and start - Allow water and low flow to settle
96. "USE THIS AND LOW FLOW FOR APPROX 1 MIN
97. "Flow stop and start - Allow water and low flow to settle
98. "USE THIS AND LOW FLOW FOR APPROX 1 MIN
99. "Flow stop and start - Allow water and low flow to settle
100. "USE THIS AND LOW FLOW FOR APPROX 1 MIN

```

factor so obtained is used to calculate the Reynolds number from the Newtonian friction factor chart. This Reynolds number is equated to $\rho \bar{V} D / \mu_t$ to give μ_t , the "turbulent viscosity". This is then assumed constant. It is clear that the disadvantage of this approach is that it does not allow the design of a pipeline directly from a knowledge of flow properties. It is in fact a scale-up procedure whereby full-scale equipment can be designed once the empirical "turbulent viscosity" has been determined for a given fluid in a similar pilot plant. Alves et al. have adopted this approach (85).

The drawback of the methods discussed above is that they do not take into account the possibility of non-Newtonian behaviour prevailing under the conditions of shear existing in turbulent flow. However, the semi-theoretical analysis of Dodge-Metzner, partially takes into account the non-Newtonian behaviour prevailing under the conditions of turbulent flow.

3.32 Scale-Up Procedures

3.321 Bowen's Method (89): Bowen has proposed a scale-up method which seems to work for the turbulent flow of any time-independent viscous or viscoelastic fluid (or drag reducing fluid) or suspension which exhibits a linear relationship in a logarithmic plot of $D\Delta P/4L$ versus $\bar{8V}/D$ under turbulent conditions. A nearly straight parallel family of curves is obtained when the turbulent flow data are plotted ($\log D\Delta P/4L$ vs $\log \bar{8V}/D$) for different pipe diameters.

A similar family of nearly straight parallel curves results when turbulent flow data obtained with Newtonian fluids are plotted in the


```

10 GOSUB 2000 'initialize channel numbers
20 L=L+1
30 IF L=0 data from which pipe=FIF.NUM
40 F=0
50 INPR "Pressure transducer channel":FRE.CH#
60 Y=0 : SUM.FRE=0
70 PRINT "PRESS ANY KEY TO CONT. "
80 A$=INKEY$ : IF A$="" THEN 1656
90 CH.NO=FRE.CH# : GOSUB 4000
100 SUM.FRE=SUM.FRE + READING(FRE.CH#)
110 Y=Y+1 : IF Y=1000 GOTO 1700
120 AVG.FRE=SUM.FRE/1000 'FREQUENCY IN Pa.s
130 INPR "flow rate units--if units=mixed then rate unit.flo=1---if units=g/
then unit.flo=2:UNIT.FLO
140 INPR "flow rate in units specified above":RATE.FLO
150 IF UNIT.FLO=1 THEN LOW.Q=RATE.FLO/10.6
160 IF UNIT.FLO=2 THEN LOW.Q=RATE.FLO/106.AVG
170 PRINT #1,USING"###.##### ":R:FIF.NUM;LOW.Q;AVG.FRE
180 LPRINT USING"###.#### ":R:FIF.NUM;LOW.Q;AVG.FRE
190 P=P+1 : P=P+1 : PRINT "no of flow rate runs taken=" :P
200 INPR "no of flow rate runs desired ":I
210 IF P=I GOTO 1590
220 L=L+1 : IF L=5 GOTO 1560 'data from pipes 1,2,3,4 and 5
230 CLOSE #1 : RETURN
240 ***** LOW FLOW SUB ENDS *****
250 ***** INITIALIZE CHANNEL NUMBERS SUB BEGINS *****
260
270
280 RESTORE 2080
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Ch	Variable	Description	Eng
#	Label		Units
0	CH 0,	Test Data for Channel 0,	V
1	CH 1,	Test Data for Channel 1,	V
2	CH 2,	Test Data for Channel 2,	V
3	CH 3,	Test Data for Channel 3,	V
4	CH 4,	Test Data for Channel 4,	V
5	CH 5,	Test Data for Channel 5,	V
6	CH 6,	Test Data for Channel 6,	V
7	CH 7,	Test Data for Channel 7,	V
8	CH 8,	Test Data for Channel 8,	V
9	CH 9,	Test Data for Channel 9,	V
10	CH 10,	Test Data for Channel 10,	V
11	CH 11,	Test Data for Channel 11,	V
12	CH 12,	Test Data for Channel 12,	V
13	CH 13,	Test Data for Channel 13,	V
14	CH 14,	Test Data for Channel 14,	V
15	CH 15,	Test Data for Channel 15,	V
16	CH 16,	Output Channel 1,	V
17	CH 17,	Output Channel 2,	V
-1			" -1 Indicates end of data"

```

20 READ CH.NO
30 WHILE CH.NO >= 0
40 FOR I = 1 TO 3
50 READ CHAN#(CH.NO, I)
60 NEXT I
70 READ CH.NO
80 WEND
90 RESTORE 2167

```

This equation suggests a constant A containing fluid density and viscosity:

$$A = \frac{0.079 (\rho)^{0.75} (\mu)^{0.25}}{(2) (8)^{1.75}} \quad (3.39)$$

the diameter D raised to an exponent term e equal to 1.6 for Newtonian fluids; and the flow function raised to an exponent term s equal to 1.8 for Newtonian fluids. The following general relationship:

$$\frac{D\Delta p}{4L} = A D^e \left(\frac{8V}{D}\right)^s \quad (3.310)$$

is thus suggested for any fluid describing straight parallel lines on the logarithmic plot of flow and stress function in the turbulent region. Bowen's purely empirical method requires turbulent flow friction factor data on at least two pipe diameters to obtain the power on the diameter.

3.322 Lord, Hulsey and Melton Scale-up procedure (92-93): A generalized turbulent flow relationship derived from the Blasius expression and suggested by Bowen (eqn. 3.310) can be rearranged as

$$\frac{\Delta p}{4L} = A D^{e-s-1} (8V)^s \quad (3.311)$$

```

166
67 DATA 0, 0.0, 10, 0.0, 10.0, -1.0, 11.0
68 DATA 1, 0.0, 10, 0.0, 10.0, -1.0, 11.0
169 DATA 2, 0.0, 10, 0.0, 10.0, -1.0, 11.0
70 DATA 3, 0.0, 10, 0.0, 10.0, -1.0, 11.0
71 DATA 4, 0.0, 10, -79.136584, 739.87258, -1.0, 11.0
72 DATA 5, 0.0, 10, -348.06426, 2665.8713, -1.0, 11.0
73 DATA 6, 0.0, 10, -756.46781, 13235.212, -1.0, 11.0
74 DATA 7, 0.0, 10, 0.0, 10.0, -1.0, 11.0
75 DATA 8, 0.0, 10, 0.0, 10.0, -1.0, 11.0
176 DATA 9, 0.0, 10, 0.0, 10.0, -1.0, 11.0
77 DATA 10, 0.0, 10, 0.0, 10.0, -1.0, 11.0
78 DATA 11, 0.0, 10, 0.0, 10.0, -1.0, 11.0
79 DATA 12, 0.0, 10, -2333.8491, 33573.58, -1.0, 11.0
180 DATA 13, 0.0, 10, -5485.072, 83428.613, -1.0, 11.0
81 DATA 14, 0.0, 10, -11843.756, 225104.18, -1.0, 11.0
182 DATA 15, 0.0, 10, 0.0, 10.0, -1.0, 11.0
190 DATA -1,

```

" -1 Indicates end of data"

```

200 READ CH.NO
202 WHILE CH.NO >= 0
203   READ X1, X2, Y1, Y2
209   SLOPE = (Y2 - Y1) / (X2 - X1)
210   CHAN(CH.NO, 1) = SLOPE
215   CHAN(CH.NO, 2) = Y1 - SLOPE * X1
218   READ CHAN(CH.NO, 3), CHAN(CH.NO, 4)
219   READ CH.NO
220 WEND
240 Range Setting
260   input range : 0 to +10 volts
274   input gain : FGL (Low inputs: 1,10,100,500)
299 RETURN

```

```

300 ***** INITIALIZE CHANNEL NUMBERS SUB ENDS *****
301 ***** A/D READING SUB BEGINS *****
302 TECMAR LABMASTER A/D Sampler

```

```

340 Inputs : CH.NO the channel you wish to sample
350 Outputs : READING the channel read (in final units)
360 ERR.NO set when any errors are detected
370 GAIN%=0
380 LM.BASE = &H710
390 OUT LM.BASE+4, 128+GAIN%
400 OUT LM.BASE+5, CH.NO
410 OUT LM.BASE+6, 0
420 IF INP(LM.BASE+4) < 128 THEN 4130
430 LOW.BYTE% = INP(LM.BASE+5)
440 HIGH.BYTE% = INP(LM.BASE+6)
450 VAR = LOW.BYTE% + HIGH.BYTE% * 256
460 IF HIGH.BYTE% > 1 AND HIGH.BYTE% < 128 THEN 4170
470 IF HIGH.BYTE% < 255 AND HIGH.BYTE% > 127 THEN 4170

```

```

480 Convert per cent full scale to final units
490 VAR = VAR / 2
500 VAR = VAR / 10^(GAIN%)
510 IF GAIN% = 3 THEN VAR = VAR * 2
520 VOLTAGE = VAR / 204.8
530 READING(CH.NO) = VOLTAGE * CHAN(CH.NO, 1) + CHAN(CH.NO, 2)
540 RETURN

```

```

550 ***** A/D READING SUB ENDS *****
560 ***** HIGH FLOW SUB BEGINS *****
570 HIGH FLOW SUB - HIGH TURBULENT DATA AND METERING DATA COLLECTION

```

$$\text{percent drag reduction} = \frac{(\Delta P_0 - \Delta P_{\text{additive}})}{\Delta P_0} \times 100 \quad (3.40)$$

Drag reduction is distinct from the decreased friction factor observed by Dodge and Metzner (80) and exhibited by purely viscous pseudoplastic fluids in turbulent flow at a given generalized Reynolds number. Friction factors for drag reducing fluids may be far below those predicted using the Dodge-Metzner correlation:

$$\frac{1}{\sqrt{f}} = \frac{4.0}{(n')^{0.75}} \log [N' \text{Re} f^{\frac{(1-n')}{2}}] - \frac{0.40}{(n')^{1.2}} \quad (3.34)$$

The drag reduction phenomenon is exhibited by many Newtonian and pseudoplastic solutions, gels, and suspensions, and is a departure from their "normal" viscous behaviour. The pseudoplastic friction factor effect correlated by Dodge and Metzner is regarded to be "normal" viscous behaviour.

Drag reduction occurs in single phase turbulent flow and may be achieved with several types of additives. Additives that have been used with success include high molecular weight polymers (94-113), solid particle suspensions (114-116), soaps (117-126), fibres (127-130) and other complex-forming mixtures that behave in a manner similar to soaps (131-132).

```

225 K$="PIF.NUM";M$="PIF.PRE" : N$="AVG.MAG" : O$="AVG.ORI" : Q$="READ.NO"
230 WRITE #1,TITLE$ : LPRINT "HIGH FLOW DATA"
235 PRINT#1,USING"\          \";Q$;K$;M$;N$;O$
236 LPRINT USING"\          \";Q$;K$;M$;N$;O$
240 GOSUB 2000 'initialize channel numbers
245 DIM A(3) : L=0 : K=0 : R=0
250 INPUT "MAG FLOW METER CHANNEL"; MAG.CHA
255 A(1)=MAG.CHA
260 INPUT "DATA FROM METERING SECTION AND WHICH PIPE ??";PIF.NUM
265 INPUT "PRESSURE CHANNEL FOR PIPE ";PIF.CHA
270 A(2)=PIF.CHA
275 INPUT "PRESSURE CHANNEL FOR ORIFICE"; ORI.CHA
280 A(3)=ORI.CHA
285 P=0 : SUMPIF.CHA=0
290 SUMORI.CHA=0 : SUMMAG.CHA=0
295 PRINT " PRESS ANY KEY TO CONT. "
300 A4$=INKEY$ : IF A4$="" THEN 5235
305 NO.CHA=3 : FOR V=1 TO NO.CHA : CH.NO=A(V)
310 GOSUB 4000 'READ GIVEN CHANNEL
315 NEXT V
320 SUMPIF.CHA=SUMPIF.CHA + READING(PIF.CHA)
325 SUMORI.CHA=SUMORI.CHA + READING(ORI.CHA)
330 SUMMAG.CHA=SUMMAG.CHA + READING(MAG.CHA)
335 P=P+1 : IF P<1000 GOTO 5240
340 AVGPRES.PIF=SUMPIF.CHA/1000 'Pa.S
345 AVG.ORI=SUMORI.CHA/1000 'Pa.S
350 AVG.MAG=SUMMAG.CHA/1000 'VOLTS
355 PRINT #1,USING "###.####^" ;R;PIF.NUM;AVGPRES.PIF;AVG.MAG;AVG.ORI
360 LPRINT USING "###.####^" ;R;PIF.NUM;AVGPRES.PIF;AVG.MAG;AVG.ORI
365 L=L+1 : R=R+1 : PRINT "NO OF FLOW RATE RUNS ALREADY TAKEN ";L
370 INPUT "NO OF FLOW RATE RUNS DESIRED ";T
375 IF L<T GOTO 5140
380 K=K+1 : IF K<3 GOTO 5490 ' data only from 3 pipes namely 3,4 and 5
385 CLOSE#1 : RETURN
390 L=0 : GOTO 5135
400 '*****A***** HIGH FLOW SUB ENDS *****
405 '*****METER CALIBRATION SUB BEGINS *****
410 OPEN "B:CALIBRAT.Dat" FOR APPEND AS#1
415 INPUT "BRIEF title of expt."; TITLE$
420 A$="READ.NO" : B$="MAS.FLO" : C$="AVG.MAG" : D$="AVG.ORI" : E$="AVG.VEN"
425 WRITE #1,TITLE$ : LPRINT "METER CALIBRATION DATA"
430 PRINT#1,USING"\          \";A$;B$;C$;D$;E$
435 LPRINT USING"\          \";A$;B$;C$;D$;E$
440 GOSUB 2000 'initialize channel numbers
445 DIM R(3) : L=0 : K=0
450 INPUT "MAG FLOW METER CHANNEL"; MAG.CHA
455 R(1)=MAG.CHA
460 INPUT "PRESSURE CHANNEL FOR ORIFICE"; ORI.CHA
465 R(2)=ORI.CHA
470 INPUT "PRESSURE CHANNEL FOR VENTURI ";VEN.CHA
475 R(3)=VEN.CHA
480 P=0
485 SUMORI.CHA=0 : SUMVEN.CHA=0 : SUMMAG.CHA=0 :
490 PRINT " PRESS ANY KEY TO CONT. "
495 A5$=INKEY$ : IF A5$="" THEN 9206
500 NO.CHA=3 : FOR T=1 TO NO.CHA : CH.NO=R(T)
505 GOSUB 4000 'READ GIVEN CHANNEL
510 NEXT T
515 SUMORI.CHA=SUMORI.CHA + READING(ORI.CHA)
520 SUMVEN.CHA=SUMVEN.CHA + READING(VEN.CHA)
525 SUMMAG.CHA=SUMMAG.CHA + READING(MAG.CHA)
530 P=P+1 : IF P<500 GOTO 9210
535 AVG.ORI=SUMORI.CHA/500 'Pa.S
540 AVG.VEN=SUMVEN.CHA/500 'Pa.S
545 AVG.MAG=SUMMAG.CHA/500 'VOLTS

```


The conductivity of W/O emulsions increases with the temperature and volume concentration of dispersed phase (i.e. water).

For a dispersion of spherical particles in a liquid at low concentrations, Wagner (134) derived the following relationship between the complex dielectric constant of the dispersion and the volume fraction:

$$\frac{\epsilon^* - \epsilon_m^*}{\epsilon^* + 2\epsilon_m^*} = \frac{\epsilon_p^* - \epsilon_m^*}{\epsilon_p^* + 2\epsilon_m^*} \quad \phi \quad (4.10)$$

Since the above equation is derived for a dilute dispersed system it does not hold for concentrated dispersions. Hanai (135-137) developed a theory for a concentrated dispersed system on the basis of Wagner's Theory. Hanai made the following assumptions.

1. A concentrated dispersed system can be obtained by successively adding infinitesimally small quantities of dispersed phase into the dispersion medium.
2. Wagner's relation (Eqn. 4.10) holds for this process with infinitesimally small change in concentration.

Based on the above assumptions, Hanai derived the following relationship:

$$\frac{\epsilon^* - \epsilon_m^*}{\epsilon_m^* - \epsilon_p^*} \left(\frac{\epsilon_m^*}{\epsilon^*} \right)^{1/3} = 1 - \phi \quad (4.11)$$

on substitution of the relations for ϵ^* , ϵ_p^* , ϵ_m^* and separating the real and imaginary parts, Eqn. (4.11) reduced to:

```
350 INPUT "FLOW RATE IN G/SEC"; MASS.FLO
360 PRINT#1, USING "###.##### "; L, MASS.FLO, AVG.MAG, AVG.ORI, AVG.VEN
365 LPRINT USING "###.##### "; L, MASS.FLO, AVG.MAG, AVG.ORI, AVG.VEN
370 L=L+1 : PRINT "NO OF FLOW RATE RUNS ALREADY TAKEN "; L
380 INPUT "NO OF FLOW RATE RUNS DESIRED "; T
390 IF L > T GOTO 9120
395 CLOSE#1
400 RETURN
410 ***** METER CALIBRATION SUB ENDS *****
```


$$\frac{K_h}{K_m} = (1 - \phi)^{3/2} \quad (4.16)$$

Eqn. (4.14) reduces to:

$$\frac{\epsilon_h}{\epsilon_m} = (1 - \phi)^{3/2} \quad (4.17)$$

Eqn. (4.15) reduces to:

$$\frac{K_h}{K_m} = \frac{\epsilon_h}{\epsilon_m} \quad (4.18)$$

From Eqns. (4.17) and (4.18):

$$\frac{K_h}{K_m} = (1 - \phi)^{3/2} \quad (4.19)$$

From Eqns. (4.16) and (4.19):

$$K_h = K_h \quad (4.110)$$

```

000 *****
010 P Plotting Routine for Graphics Monitor
020
030
040 plots curves of data in the X-Y co-ordinates
050
060 Required
070 x.pts() . . . . . x values
080 y.pts() . . . . . y values
090 N . . . . . number of points to be plotted
100
110 Optional
120 TITLE$ . . . . . title of graph (max = 35 chars)
130 X$ . . . . . x-axis label (max = 35 chars)
140 Y$ . . . . . y-axis label (max = 20 chars)
150 CONNECT . . . . . = -1 if points are to be connected
160 RE.PLOT . . . . . = -1 if plot is to be added to graph
170 GRID . . . . . = -1 if a grid is to be drawn
180 XMIN . . . . . minimum x-axis value
190 XMAX . . . . . maximum x-axis value
200 YMIN . . . . . minimum y-axis value
210 YMAX . . . . . maximum y-axis value
220
230 Returned
240 ERR.NO . . . . . error encountered
250 1 = zero length axis
260 2 = error detected in subroutine
270 save program and consult J.Allen
280 Variables
290 I . . . . . general counter
300 ORIGIN$ . . . . . origin of graph
310 PLOT.COLOR . . . . . color of current plot
320 TICK$ . . . . . location of next tick mark
330 X . . . . . next x value to plot in screen units
340 X.EXP% . . . . . log(x) base 10
350 X.SCALE . . . . . X (screen units) = X.pts() * X.SCALE
360 Y . . . . . next x value to plot in screen units
370 Y.EXP% . . . . . log(x) base 10
380 Y.SCALE . . . . . Y (screen units) = Y.pts() * Y.SCALE
390
95 NOTE: If the scale on the axes is not specified (via XMAX XMIN YMAX YMIN)
96 then the subroutine calculates them by scanning the array of data.
97 If the routine is called initially with a single data point then
98 it is impossible for the routine to determine a scale. The scale
99 must be specified by the caller if a single point is used.
00 DEF FNLOG10(X) = LOG(X)/LOG(10) ' log for base 10
01 IF RE.PLOT THEN GOTO 7100 ' skip screen setup
02 CLS
03 SCREEN 1,0,0,0
04 ORIGIN$ = "C2 BM49,176"
05 KEY OFF
06 COLOR 0,0
07
08 Find Limits of axes
09
10 IF XMAX <> XMIN THEN GOTO 6570 'user chosen X axis
11 XMAX = XMIN = X.PTS(1)
12 FOR I = 1 TO N
13 IF XMAX < X.PTS(I) THEN XMAX = X.PTS(I)
14 IF XMIN > X.PTS(I) THEN XMIN = X.PTS(I)
15 NEXT I

```

where

C_D = discharge coefficient which is a function of Reynolds number ($= \rho D_0 \bar{V} / \mu$)

A_0 = throat (or orifice) cross-sectional area

$R = D_0/D$ = ratio of throat (or orifice) diameter to pipe diameter

Differential Pressure Meters for Emulsions:

If it is assumed that the emulsions behave as single phase liquids, the following equation (i.e. homogeneous flow model) can be used to determine the mean flow rate through the meter.

$$W = \frac{C_D A_0}{\sqrt{1 - R^4}} \sqrt{2\rho \Delta p}$$

where

ρ = mean density of the mixture

C_D = discharge coefficient

Discharge Coefficient Versus Reynolds Number

The principal difference between single phase flow and emulsion flow is the choice of viscosity to be used in the Reynolds number. The following methods will be used to correlate discharge coefficient versus Reynolds number in the present study.

(1) Viscosity of dispersion medium: Caldwell and Babbitt (84) in their work on pipeline flow of sewage sludges and slurries used the viscosity of the dispersion medium (in the Reynolds number) to

```

570 IF YMAX <> YMIN THEN GOTO 6630 'user chosen Y axis
580 YMAX = YMIN = Y.PTS(1)
590 FOR I = 1 TO N
600 IF YMAX < Y.PTS(I) THEN YMAX = Y.PTS(I)
610 IF YMIN > Y.PTS(I) THEN YMIN = Y.PTS(I)
620 NEXT I
630
640 PLOT axes
650
660 DRAW ORIGIN$ ' move to origin
670 DRAW "U160 L7 d165 R267 U5 L260" ' outline axes
680 PFAINT STEP(-1,-1),2,2 ' fill in axes
690
700 Plot Scale on Axes
710
720 FOR I = 0 TO 10 'along x axis
730 X = I * 24 + 50
740 IF GRID THEN TICK$ = "BM" + STR$(X) + ",15 M" + STR$(X) + ",190"
750 ELSE TICK$ = "BM" + STR$(X) + ",180 M" + STR$(X) + ",190"
760 DRAW TICK$
770 NEXT I
780
790 FOR I = 0 TO 10 'along y axis
800 Y = 175 - I * 16
810 IF GRID THEN TICK$ = "BM37," + STR$(Y) + " M290," + STR$(Y)
820 ELSE TICK$ = "BM37," + STR$(Y) + " M42," + STR$(Y)
830 DRAW TICK$
840 NEXT I
850
860 Put Scale on axes
870
880 IF XMAX-XMIN = 0 OR YMAX-YMIN = 0 THEN ERR.NO = 2: RETURN
890 X.EXP% = FNLOG10(XMAX-XMIN)
900 Y.EXP% = FNLOG10(YMAX-YMIN)
910 FOR I = 0 TO 5
920 LOCATE 24, 6*I + 5
930 PRINT USING"##.#"; (XMIN + I*((XMAX-XMIN)/5))/ 10^X.EXP%;
940 LOCATE 22 - I*4,2
950 PRINT USING"##.#"; (YMIN + I*((YMAX-YMIN)/5))/ 10^Y.EXP%;
960 NEXT I
970 LOCATE 25,34: PRINT "E";:PRINT USING "##";X.EXP%;
980 LOCATE 23,1 : PRINT "E";:PRINT USING "##";Y.EXP%;
990
1000 Plot Titles
1010
1020 TITLE$ = MID$(TITLE$,1,35) ' truncate titles
1030 X$ = MID$(X$,1,35)
1040 Y$ = MID$(Y$,1,20)
1050 LOCATE 1,(20-INT(LEN(TITLE$)/2)): PRINT TITLE$;
1060 LOCATE 25,(20-INT(LEN(X$)/2)): PRINT X$;
1070 CHARS = (12-INT(LEN(Y$)/2))
1080 FOR I = 1 TO LEN(Y$)
1090 LOCATE I + CHARS, 1
1100 PRINT MID$(Y$,I,1);
1110 NEXT I
1120
1130 Plot Data Points
1140
1150 IF XMAX-XMIN = 0 OR YMAX-YMIN = 0 THEN ERR.NO = 1: RETURN 'bad scales
1160 X.SCALE = (XMAX - XMIN) / (510 - 70)
1170 Y.SCALE = (YMAX - YMIN) / (175 - 15)
1180 PLOT.COLOR = PLOT.COLOR + 1
1190 IF PLOT.COLOR > 3 THEN PLOT.COLOR = 1
1200 FOR I = 1 TO N
1210 X = X.PTS(I); Y = Y.PTS(I)

```

Chapter 5

EXPERIMENTAL APPARATUS AND CALIBRATION

The experimental apparatus descriptions given in this Chapter pertain to Tasks 1, 2, 3, 5, 6 and 7 of the contract.

5.1 General Description of Flow Loop Apparatus

The emulsion flow measuring apparatus consists of a loop in which emulsions may be prepared and recycled through various sizes of pipelines and instruments. A schematic diagram and general views of the experimental apparatus are shown in Figures 5.1, 5.2 and 5.3. It consists of two centrifugal pumps (Hayward Gordon-model AA6 and Hayward Gordon model IFLA) both of them having a by-pass to regulate the flow; two-S.S. mixing tanks of capacity 260 U.S. gals. and 60 U.S. gals.; two Philadelphia portable mixers; five-S.S. straight pipe test sections; metering test section consisting of orifice plate meter, venturimeter, magnetic flow meter and a conductivity meter; a temperature controller with heating (cooling) coil in the main tank; S.S. pressure tank containing water to purge the pressure lines and taps; several validyne transducers; manometers; various thermocouples and a measuring tank to measure the flow rates. An IBM personal computer is used to collect data from various electrical signal transducers (see below for description of the data acquisition system).

```

210 IF X < XMIN THEN X = XMIN
220 IF X > XMAX THEN X = XMAX
230 IF Y < YMIN THEN Y = YMIN
240 IF Y > YMAX THEN Y = YMAX
250 X = 50 + INT( (X - XMIN) / X.SCALE )
260 Y = 175 - INT( (Y - YMIN) / Y.SCALE )
270 IF NOT CONNECT THEN PSET(X,Y),PLOT.COLOR:GOTO 7300
280 IF CONNECT AND I > 1 THEN LINE (X.OLD,Y.OLD)-(X,Y),PLOT.COLOR
290 X.OLD = X
300 Y.OLD = Y
310 NEXT I
320 RETURN

```